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COMBUSTION TESTS OF BLACK-POWDER SUBSTITUTES
FOR THE HAND-HELD SIGNAL FLARE

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JANUARY 1991

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13. ABSTRACT (Maximum 200 words) Replacement of the black powder in hand-held signal flares with energetic materials of more consistent combustion properties is being studied. The black powder is currently used in both pressed and granular states to provide for the functions of ignition, propulsion, timing, payload expulsion, and ignition of the illuminant. The laboratory work described in this report is intended to provide a rational basis for evaluating candidate materials to replace black powder in these various functions. Linear burn rates of pressed materials were determined over the pressure range of 2 MPa to 4 MPa for Goex black powder, Pyrodex RS, and four black-powder substitutes. Fixtures were designed and instrumented to simulate those flare functions requiring granular forms of the energetic material. Temperature and pressure data from these fixtures permit a comparison of the performance of the candidate substitutes relative to black powder in the motor-ignition and payload-expulsion phases of the flare operation.				
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1. INTRODUCTION

This work is part of the task to find energetic materials to replace black powder in the hand-held signal flare (e.g., SIGNAL, ILLUMINATION, GROUND RED STAR, CLUSTER, M158). This device is a percussion-fired item in which a granulated black-powder charge ejects a small rocket from a hand-held launcher. The motor of the rocket consists of hollow cylinders of pressed black powder which are ignited during launch. As the motor charge is consumed, a delay pellet of pressed black powder burns along a connecting passage. When this delay charge is consumed, a granulated black-powder expelling charge is ignited to deploy and ignite the flare mixture. This device utilizes the recognized positive characteristics of black powder such as small pressure exponent of burn rate, ease of ignitability, and utility in the ignition of other materials. Conventional black powder, however, exhibits an undesirable degree of variability in its performance, a trait generally attributed to its charcoal component. Consequently, one direction of the effort has been to replace the charcoal in black powder with a single organic compound to reduce the uncertainty in pyrolyzed natural wood composition.¹

Four of these charcoal-substitutes were utilized by Picatinny Arsenal to fabricate four black-powder substitute materials. These items were in the form of fine powder for pressing into sticks. The charcoal-substituted materials were not provided in Class 5 granular form for testing in the granular-charge fixtures to be described. In addition, Pyrodex RS, manufactured by the Hodgdon Powder Co. was supplied by the manufacturer for testing. This material is granular with a particle size just under Class 5 and could be used to form pressed sticks as well as compared with black powder in the loose-charge combustion tests. The linear burning rates of the pressed materials over the operating pressures of the flare were measured along with pressed Goex black powder as a baseline. The performance of black powder and Pyrodex RS in the granular-charge applications were tested in special fixtures designed to mimic the internal geometry of the flare. Pressures and temperatures produced by these two materials were measured in these fixtures. The resulting data provide a basis for judging the probable performance of these materials relative to black powder with respect to the various functions of energetic material in the flare.

2. FORMULATIONS

The black powder is assumed to be a standard potassium nitrate (75%), sulfur (10%), charcoal (15%) formulation. The substitutes replace the charcoal with the chosen organic compound. The four compounds chosen for this effort are phenolphthalein, fluorescein, anthraflavic acid, and isophthalic acid. The formulation of Pyrodex is proprietary information.

3. PRESSED-MATERIAL COMBUSTION TESTS

The samples were pressed in custom dies from SPECAC (Kent, England). The die bore is 6 mm in diameter by 65 mm long. A Carver Laboratory Press with a 12 ton hydraulic jack was used to apply force to the die pistons. The press was calibrated

against a force ring from Morehouse Instrument Co. at 13350 N (3000 pounds) and 17800 N (4000 pounds).

Initial pressing tests were performed with potassium nitrate as a simulant for the black powder. Dry pressing of the material proved to be a problem. The samples would chatter during pressing from the die and pulverize or break into short pieces. Previous work (Sasse) reported success at low percentile water addition. A light application of WD40 to the die and 2 wt % water to the powder resulted in samples that were generally removed from the die easily and were of uniform translucent appearance. A drop of moisture was expressed from the die during the pressing. The potassium nitrate sample pressed with 13350 N (3000 pounds) resulted in a sample calculated as 99.3% of crystal density with no correction for retained water and no effort at forced drying. The energetic material was moistened in a plastic bag using an atomizer to spray the powdered sample. The bag was shaken to distribute the droplets thru the sample. Approximately 2% water was added. The moisture was allowed to diffuse thru the sample enclosed in the bag for at least an hour before press runs.

The moistened powder was loaded into the die with manual compaction. One plunger was in the die in a fixed position to determine the quantity of material used. The die assembly was completed and it was loaded into the press. The force on the die plunger was raised to 17800 N (4000 pounds) over a time period of thirty seconds and held there for the remainder of a five minute cycle. After the die was removed from the press and disassembled, a large mechanical press was used to push the sample from the die. The samples all had a diameter of about 6 mm and lengths in the range 25 - 31 mm. The pressing force applied to the die and the resulting average densities and standard deviations for the samples are given in Table 1. In most cases a droplet of water was expressed from the die during pressing. Noticeably less water was expressed with the black powder samples.

The samples were burned in a uniaxial mode in a windowed chamber pressurized to the desired level with nitrogen. The samples were oriented with axis vertical and ignited at the top end with a hot wire. The burn was recorded with video equipment and the rate data were obtained from digitized position versus time data, which were fitted with a linear least-squares line routine. The rate datum was obtained as the slope of this line. The rate data for the various pressures were fitted to the usual power-law expression and summarized in Table 2. The burn rate data plots are presented in Figure 1 for a pressure range of 2 MPa to 4 MPa (300 psi to 600 psi). As shown in the plots, the anthraflavic acid substitute is nearest to the Goex in burn rate over this pressure range.

Figure 2 shows an example of the position/time plot from which the rate datum is obtained. The agreement of the points and the line gives a notion of the steadiness of the burn. In general, the burns of all the substitutes and the Goex samples were very regular after the ignition perturbation smoothed out. The Pyrodex samples were somewhat more irregular in their steadiness.

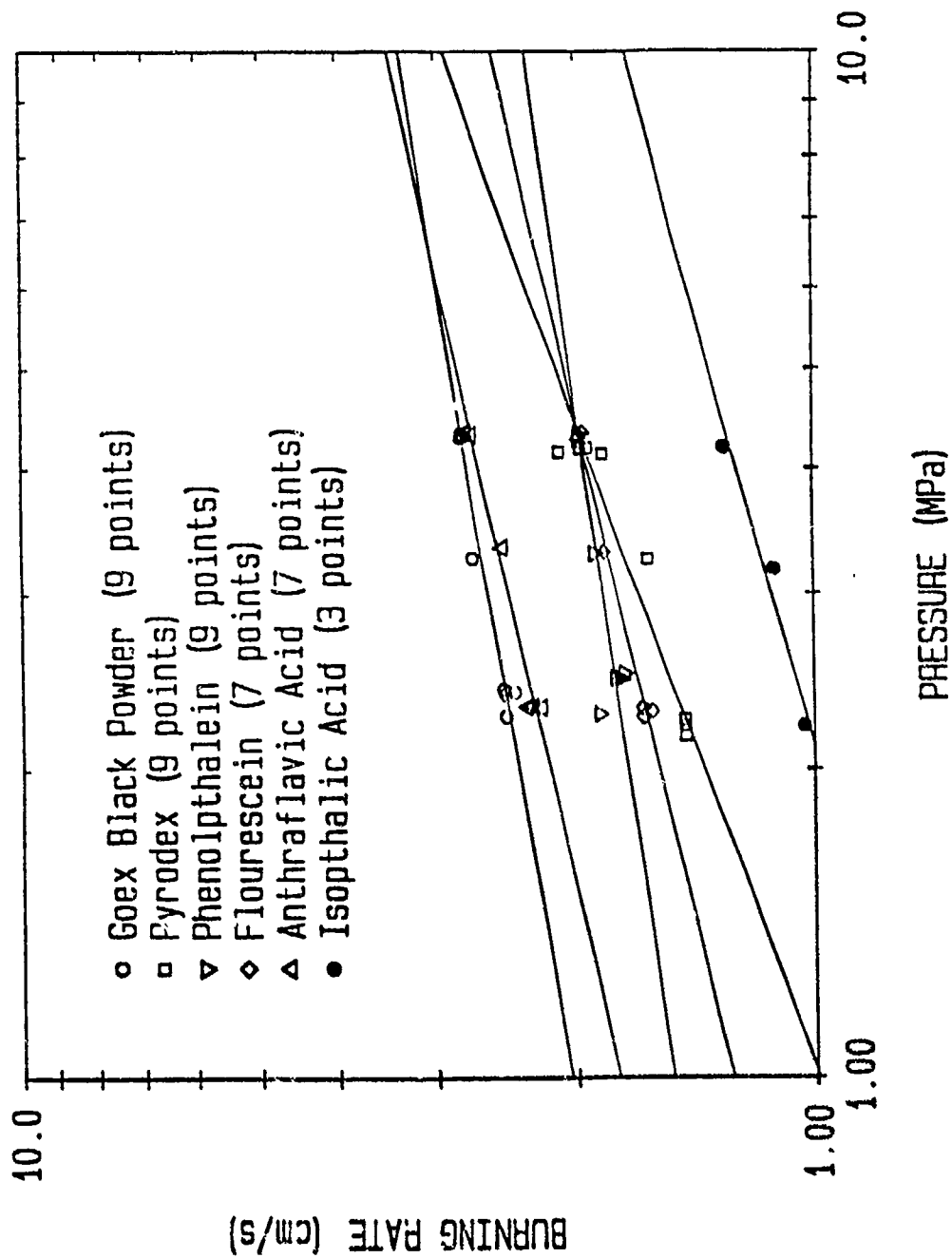


Figure 1. Pressed-Stick Burning Rates

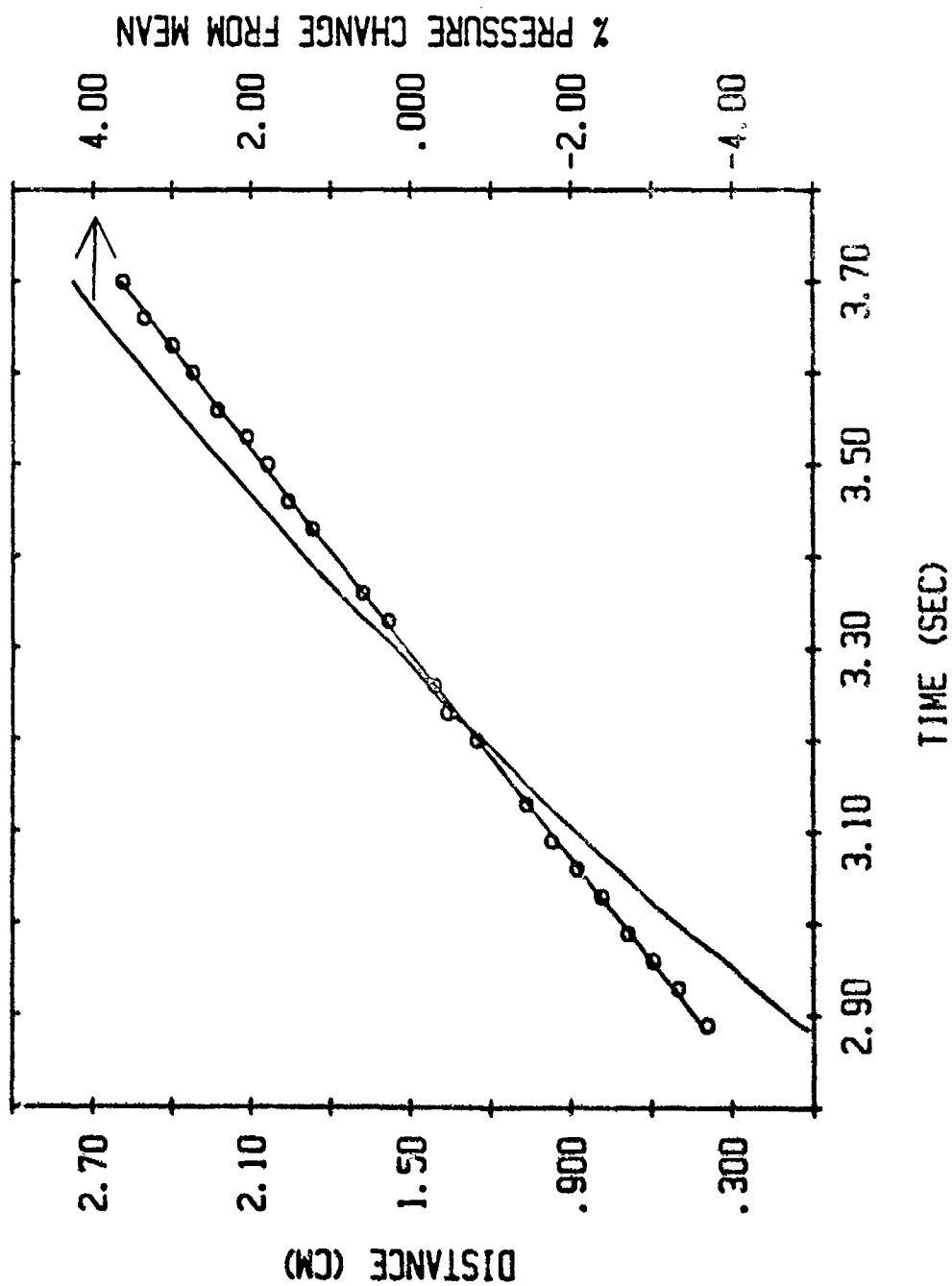


Figure 2. Anthraflavic Acid: 4.32 MPa Average

Table 1. Measured Densities of Pressed Samples			
MATERIAL	DIE FORCE (N)	DENSITY (g/cc)	# SAMPLES
Phenolphthalein	17800	1.8871 \pm .0049	14
Phenolphthalein	8900	1.8656 \pm .0004	2
Fluorescein	17800	1.9546 \pm .0076	20
Anthraflavic Acid	17800	1.9601 \pm .0057	20
Isophthalic Acid	17800	1.8921 \pm .0063	20
Goex BP	17800	1.9294 \pm .0062	20
Goex BP	13350	1.9135 \pm .0085	2
Goex BP	8900	1.9161 \pm .0028	3
Pyrodex	17800	1.8525 \pm .0055	10

Table 2. Parameters of Burning-Rate Law $r(\text{cm/s}) = b P^n(\text{MPa})$		
MATERIAL	COEFFICIENT b	EXPONENT n
Goex Black Powder	2.014	0.2126
Phenolphthalein	1.512	0.1828
Fluorescein	1.273	0.2997
Anthraflavic Acid	1.773	0.2876
Isophthalic Acid	0.7727	0.3456
Pyrodex	0.9940	0.4680

4. GRANULAR-MATERIAL COMBUSTION TESTS

Laboratory-fixtured tests were desired in order to burn the materials under conditions approximating those found in the hand-held signal flare. Two fixtures were made. One fixture was designed to examine the expelling-charge combustion phase. The other fixture was made to examine the rocket-motor igniting-charge combustion.

The igniting-charge test fixture, Figure 3, examines a small high velocity jet impinging against the material to be ignited here simulated by the heat sensor, Figure 4.

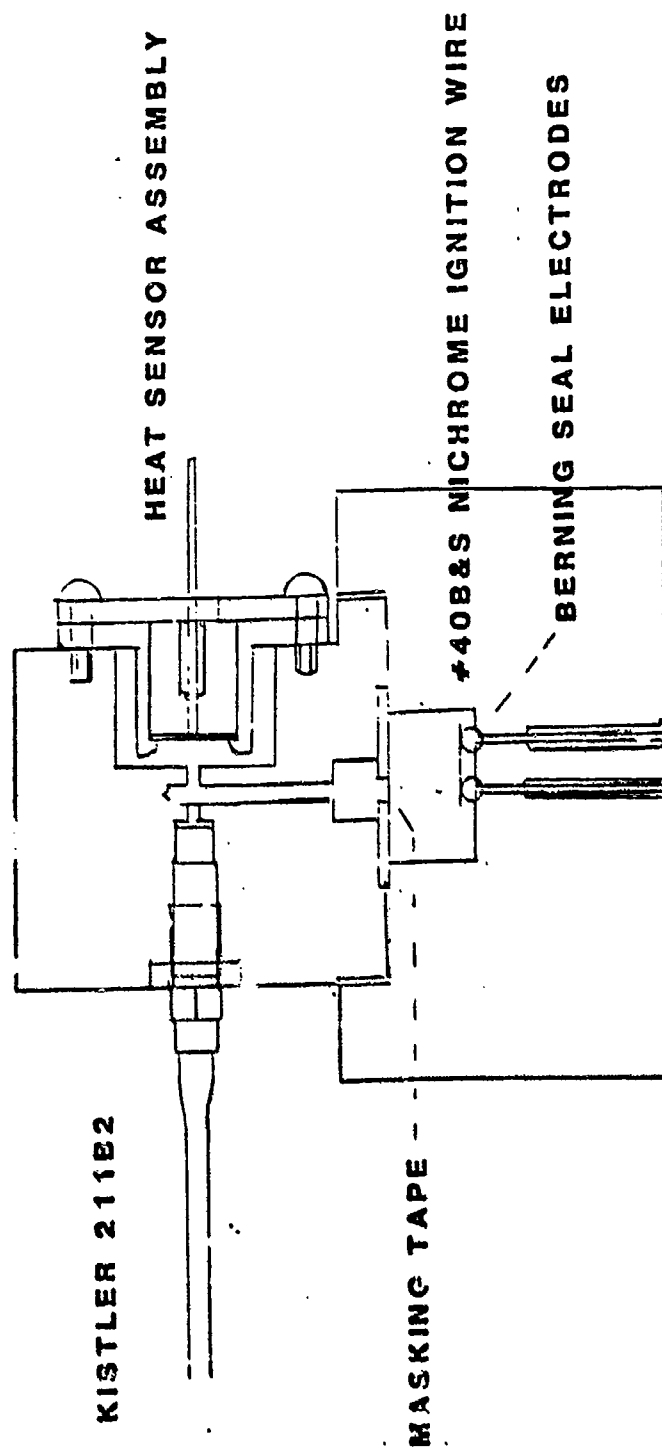


Figure 3. Igniting-Charge Test Fixture

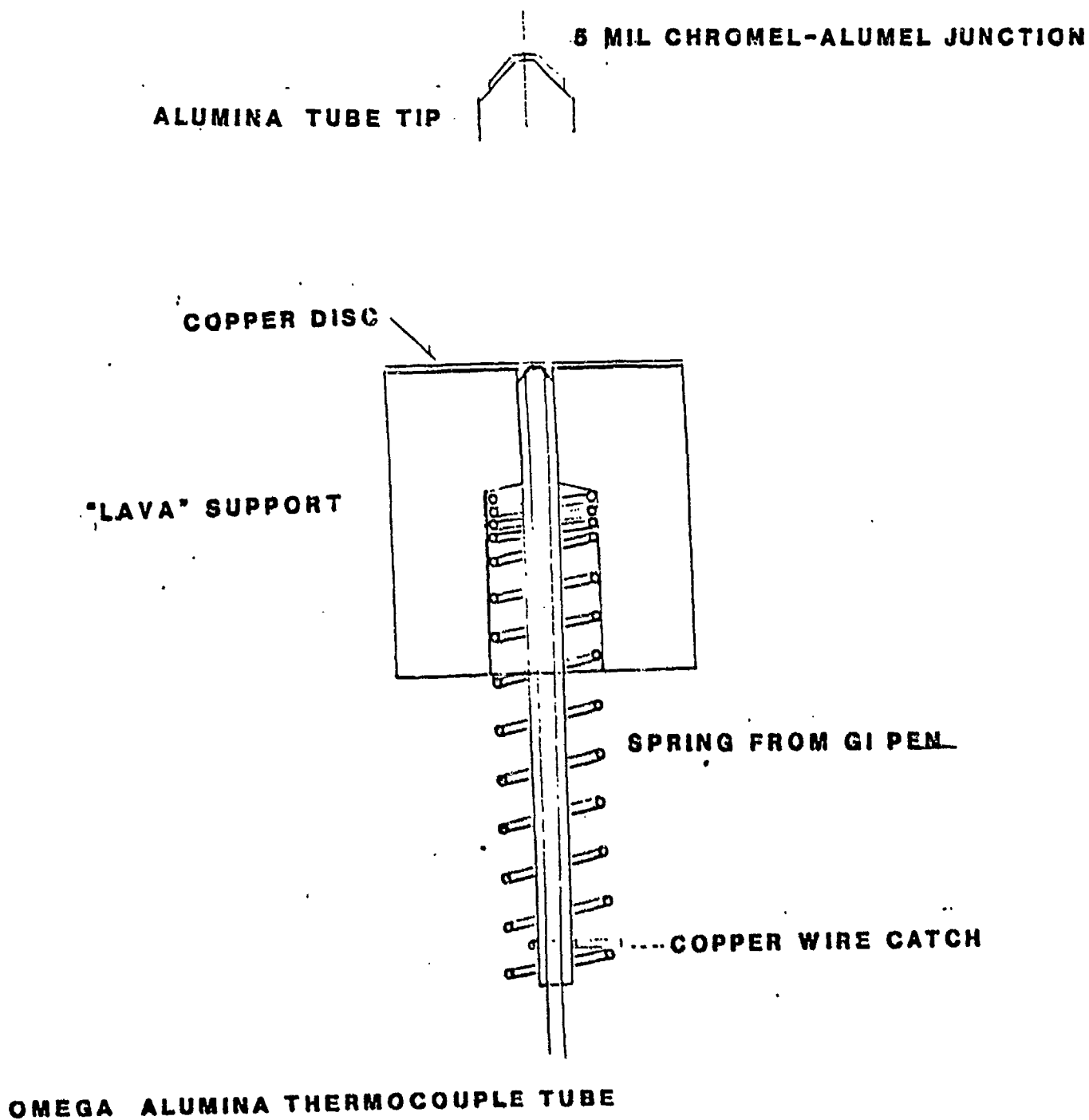


Figure 4. Heat Sensor, Thermocouple Assembly

The expelling-charge fixture, Figure 5, is intended to examine the pressurization and heat transfer during the payload-deployment phase. The venting of the combustion products is adjustable by using shims of various thickness at the "top" of the chamber. The shims used for the test sequence reported here were 0.254 mm (0.010 in.) stainless steel.

Pressure instrumentation consisted of a Kistler Pressure Transducer Model 211B2 with a Piezotron 5120 Coupler.

Devices were desired which would examine the relative heat transfer from the propellant products under several of the conditions encountered in the flare. Wall heat transfer, as in the expelling charge fixture would be characterized by relatively low velocity combustion products. For the ignition fixture, a jet of particle loaded combustion products would impact a surface at normal incidence. The heat sensors for both fixtures consisted of discs of copper exposed to the combustion gases on one side. The copper disc is supported by a ceramic cylinder with a 1.6 mm (1/16 inch) hole along the axis. A 0.127 mm (0.005 inch) wire chromel/alumel thermocouple in a 1.6 mm (1/16 inch) alumina thermocouple tube is inserted in the ceramic support and butts against the atmospheric pressure side of the copper disc. A ballpoint-pen spring was used to tension the thermocouple tube against the copper disc.

For the ignition charge test fixture 12.7 mm (1/2 inch) diameter copper discs, 0.64 mm thick were used in the heat sensor. The position of the thermocouple is troublesome in this fixture. The jet is small in diameter and position shifts of the thermocouple off the jet centerline alters the sensed temperature rapidly. Marking of the components of the heat sensor allows the parts to be assembled in the same position each time. In tests with this sensor, an erosion dimple at the jet impact point that is about half the thickness of the copper occurs for the black-powder shots. For the Pyrodex shots the erosion pit is somewhat shallower and somewhat broader.

For the sensor in the expelling-charge fixture, the copper disc is 0.12 mm thick. Variations in the shape and peak values of the temperature curves in these tests are believed to be due to the combustion product droplets flowing across the face of the disc. Upon disassembly of the sensor after a black-powder shot, residue in the shape of rivulets was observed. For the Pyrodex shots some of the examinations showed very little residue on the sensor face.

Masking tape was used over the hole between the two major parts of the chambers to provide initial charge confinement. This tape, to some degree, mimics the kraft paper used in the signal flare. The masking tape in the expelling-charge fixture was slit at one edge segment of the hole so that the tape would flop open in a repetitive fashion away from the heat sensor.

Pressure and temperature data were recorded on a Nicolet 4094 B2 scope using a 4851 plug-in.

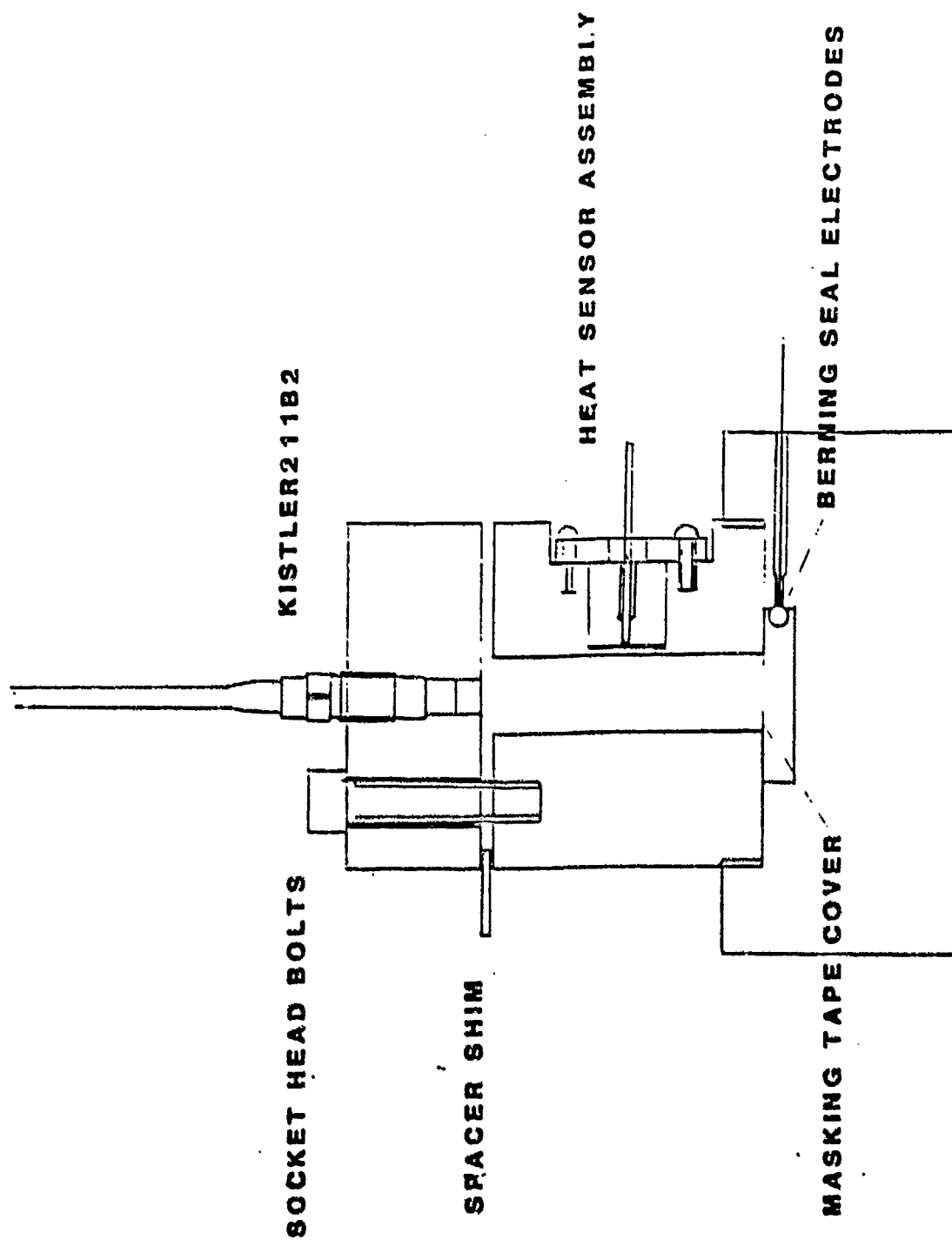


Figure 5. Expelling-Charge Test Fixture

The charge of powder for the reported tests was set at 1.000 grams. Ignition was achieved with #40 B&S gauge nickel wire spot welded into position atop the Berning seals electrodes. It was found necessary to tension this wire during spot welding in order to obtain repeatability in the pressure traces. For the expelling charge fixture, the chamber axis had to be oriented horizontally, with the ignition wire down, to obtain repeatable ignition.

Based upon an examination of the data from the ignition fixture tests, it is recommended that the charge of powder be reduced and the fixture be redesigned in order to lower the pressures obtained before additional tests are performed. As tested, the pressures obtained were nearly at the damage point of the gage. An improved seal between the fixture segments would probably make the pressure traces more reproducible.

Table 3 lists a summary of the peak pressure and temperature data obtained in the ignition fixture and the expelling charge fixture for Goex Class 5 black powder and for Pyrodex RS powder. Samples of the substitute candidate powders were not available in the proper granulation for tests in the flare fixtures. The granulation of the Pyrodex RS powder is visibly finer than that of the Goex Class 5 black powder. The detailed data for these shots is presented in Appendix A.

Table 3. Average Peak Pressure and Temperature Changes in Fixture Tests			
FIXTURE	MATERIAL	ΔP (MPa)	ΔT (°C)
Ignition	Goex Black Powder	40.22 ± 1.41	716 ± 49
Ignition	Pyrodex RS	51.21 ± 1.18	567 ± 5
Expelling-Charge	Goex Black Powder	9.70 ± 1.25	96 ± 31
Expelling-Charge	Pyrodex RS	6.75 ± 0.93	78 ± 17

8. CONCLUSIONS

Based upon the burn rate data, the anthraflavic acid substitute might function as a direct replacement for the Goex black powder in the 2 to 4 MPa range expected in the flare. The material should be granulated to the appropriate size for testing in the fixtures. The phenolphthalein substitute with the lowest pressure exponent obtained in these tests deserves testing with probable granulation adjustment.

The isophthalic acid mixture as tested in the strand burner produced large droplets of residue that tended to plug the vents of the chamber. This is probably due to the much

lower burn rate of the material. This material may be of special interest as an igniter material in other applications.

The Pyrodex gave slightly higher pressures in the ignition fixture tests and lower pressures in the expelling charge fixture.

Before additional tests are performed with the ignition fixture, it is recommended that the charge of powder be reduced and that the chamber be modified to lower the peak pressures that are obtained.

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REFERENCES

1. S. Wise, R.A. Sassé, and H.E. Holmes, "Organic Substitutes for Charcoal in "Black Powder" Type Pyrotechnic Formulations", BRL Technical Report ARBRL-TR-02569, July 1964.

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APPENDIX DATA FROM FIXTURE COMBUSTION TESTS

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APPENDIX: DATA FROM FIXTURE COMBUSTION TESTS

Table A-1. Ignition-Fixture Data				
MATERIAL	RUN #	ΔP (MPa)	ΔT (°C)	DISC MASS (g)
Goex BP	48	41.0	771	0.725
Goex BP	49	41.8	686	0.731
Goex BP	50	37.1	697	0.733
Goex BP	51	41.0	634	0.725
Goex BP	52	40.3	762	0.741
Goex BP	53	39.8	746	0.730
Pyrodex RS	59	---	562	0.750
Pyrodex RS	60	52.2	570	---
Pyrodex RS	61	48.8	570	0.736
Pyrodex RS	62	51.7	562	0.728
Pyrodex RS	63	52.4	565	0.745
Pyrodex RS	64	50.8	574	0.738
Pyrodex RS	65	51.4	---	0.745

Table A-2. Expelling-Charge Fixture Data

MATERIAL	RUN #	ΔP (MPa)	ΔT ($^{\circ}C$)
Goex BP	34	9.30	100
Goex BP	35	11.22	132
Goex BP	36	9.51	128
Goex BP	37	10.81	54
Goex BP	73	7.67	68
Pyrodex RS	66	6.14	90
Pyrodex RS	67	8.18	73
Pyrodex RS	68	6.36	63
Pyrodex RS	69	7.90	82
Pyrodex RS	70	6.04	55
Pyrodex RS	71	5.96	106

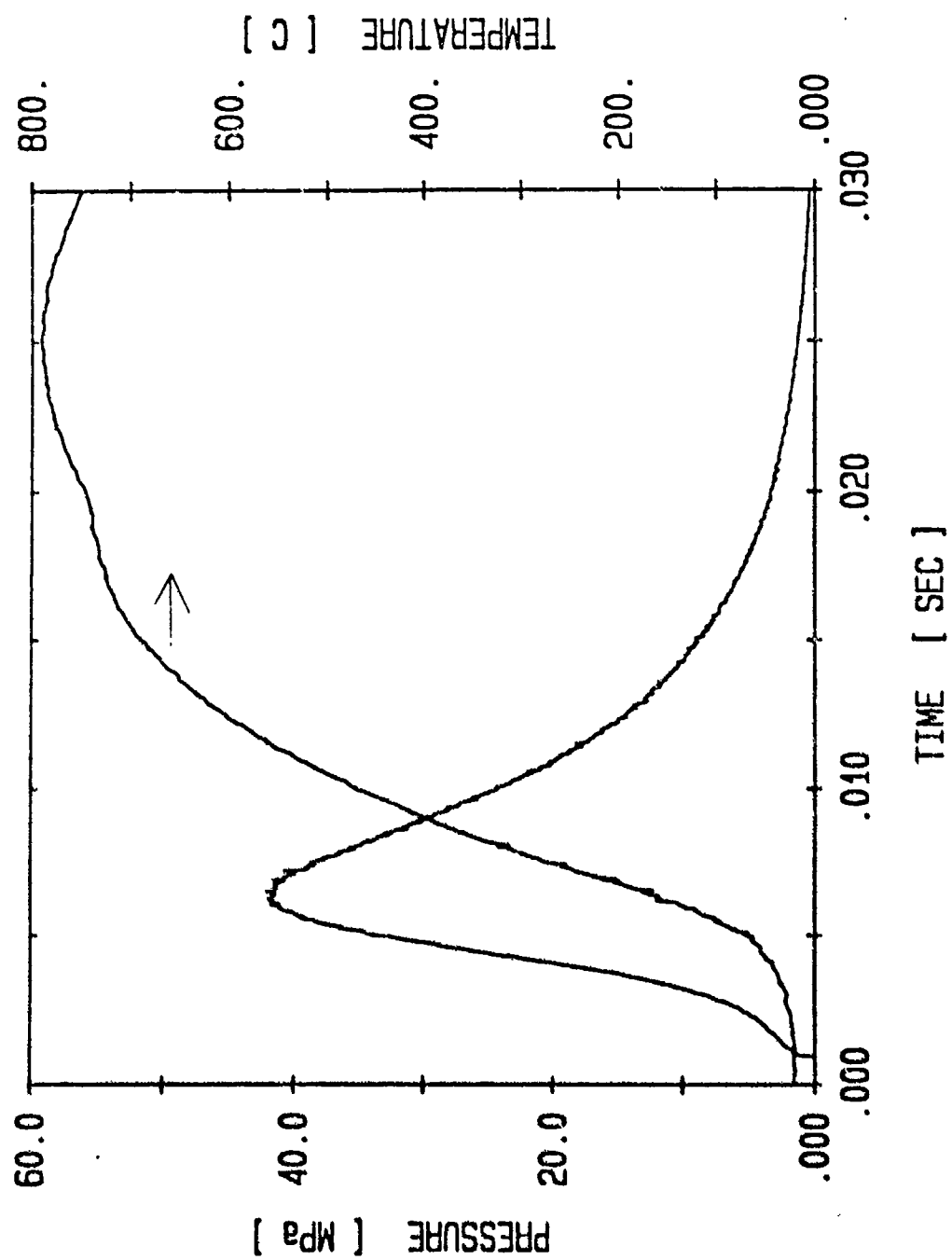


Figure A-1. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 48]

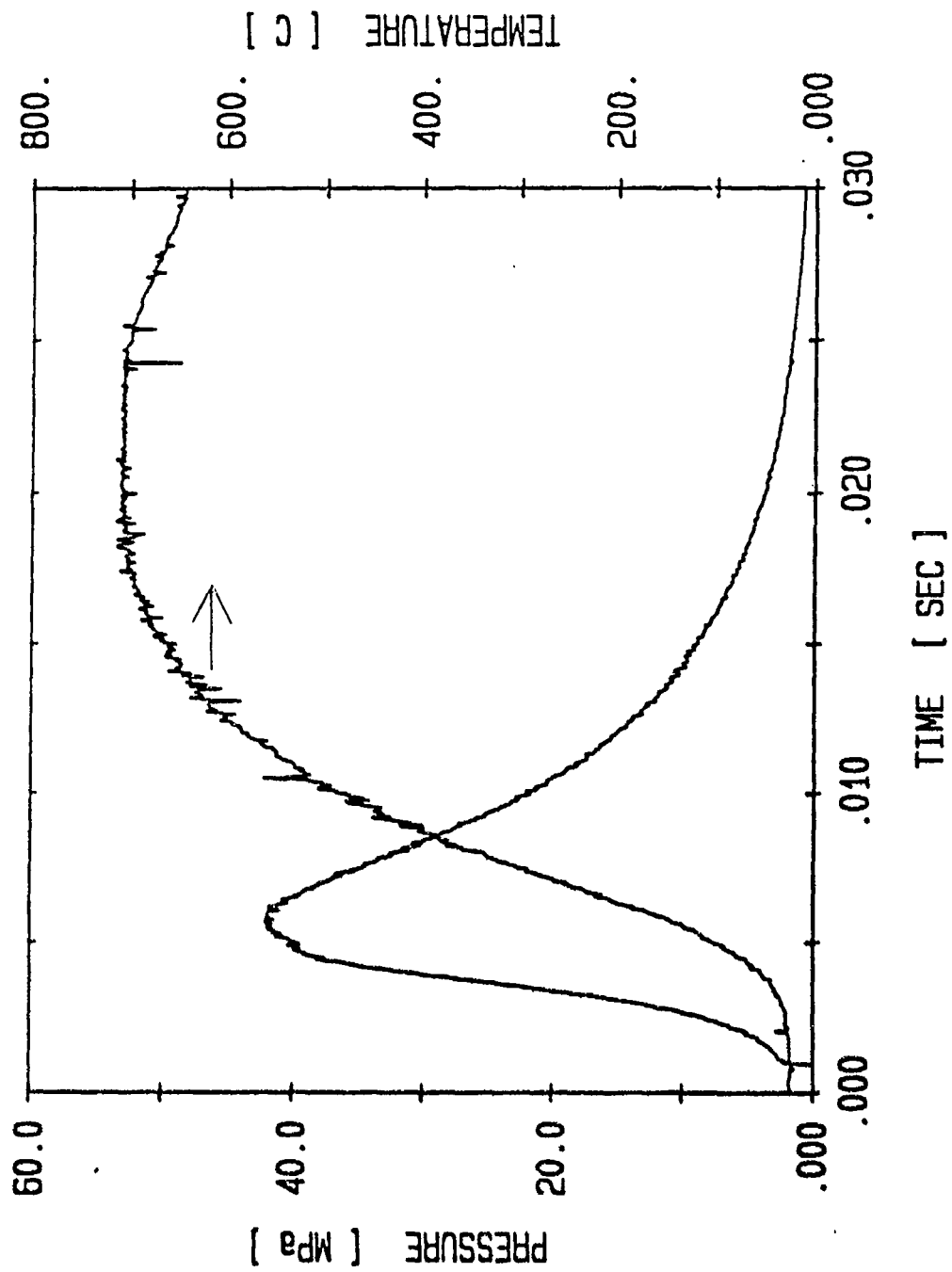


Figure A-2. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 49]

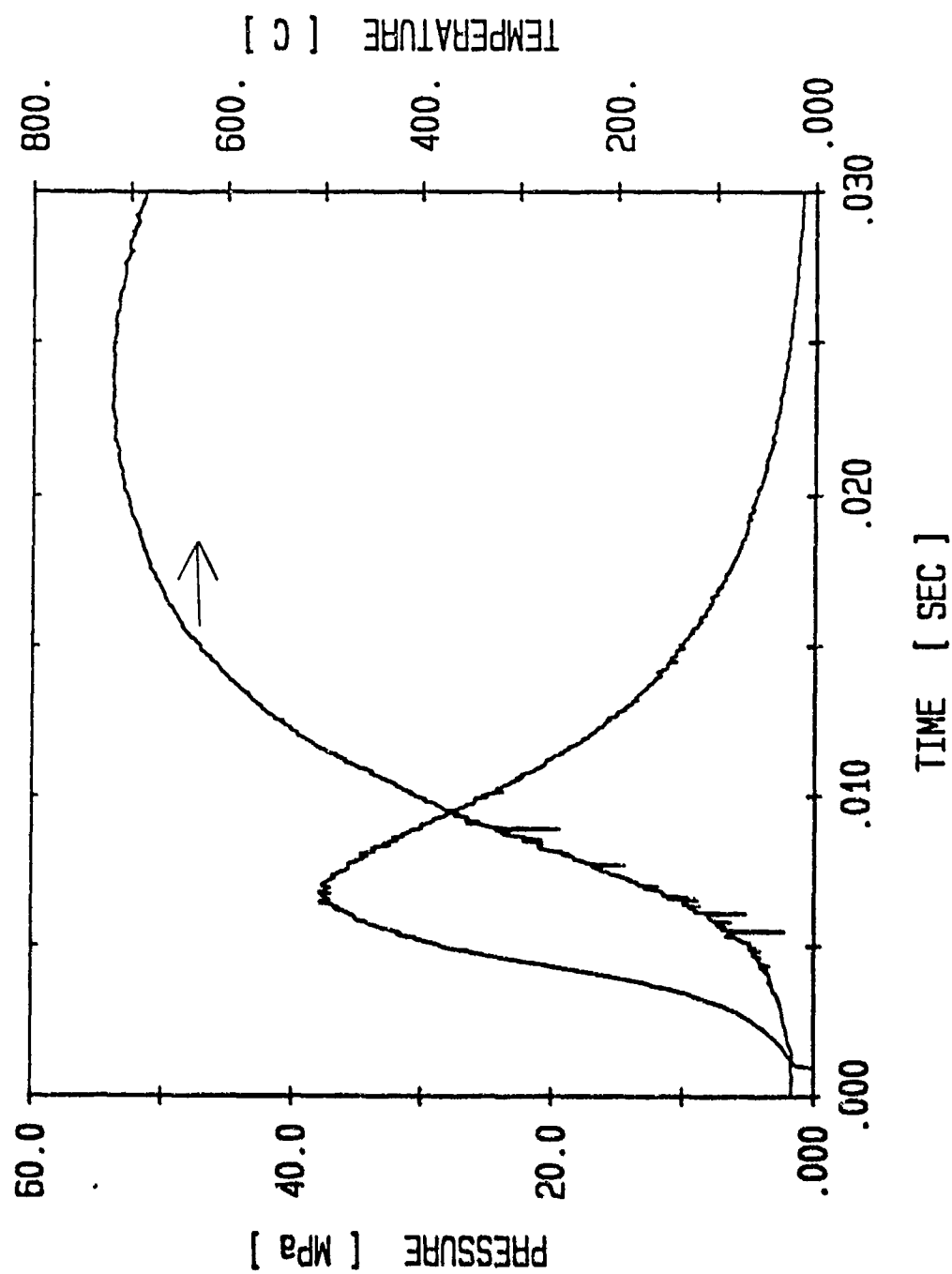


Figure A-3. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 50]

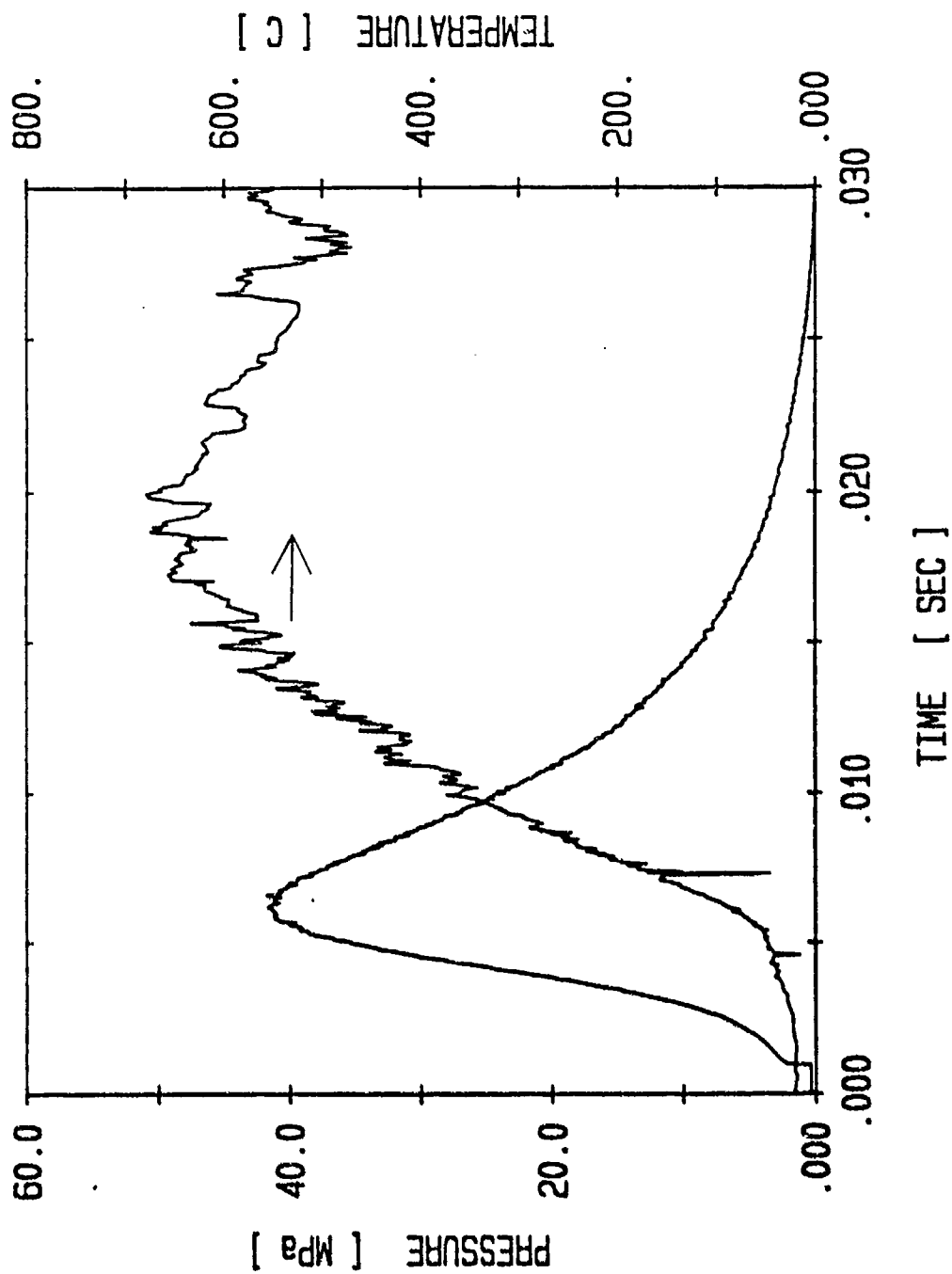


Figure A-4. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 51]

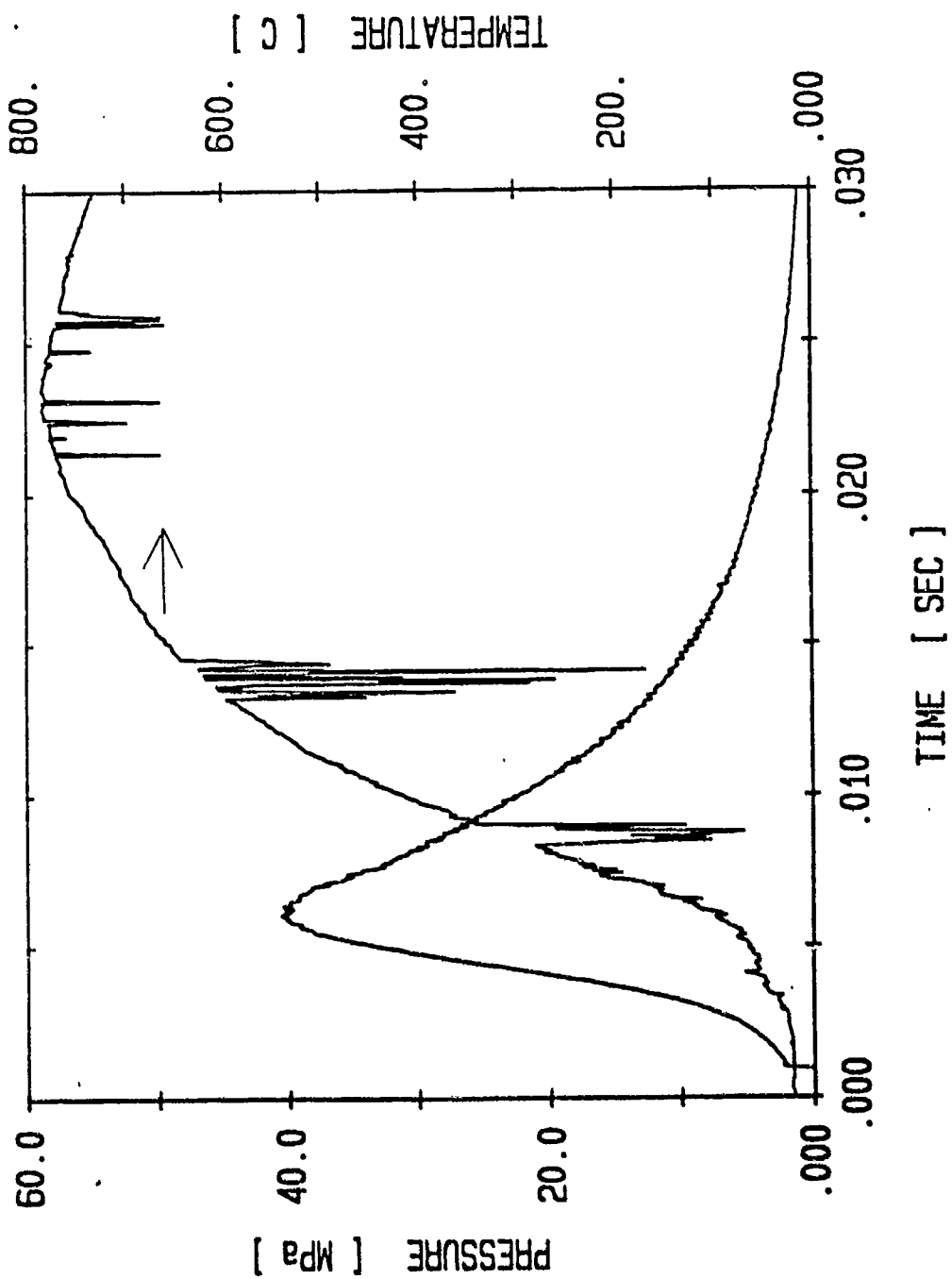


Figure A-5. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 52]

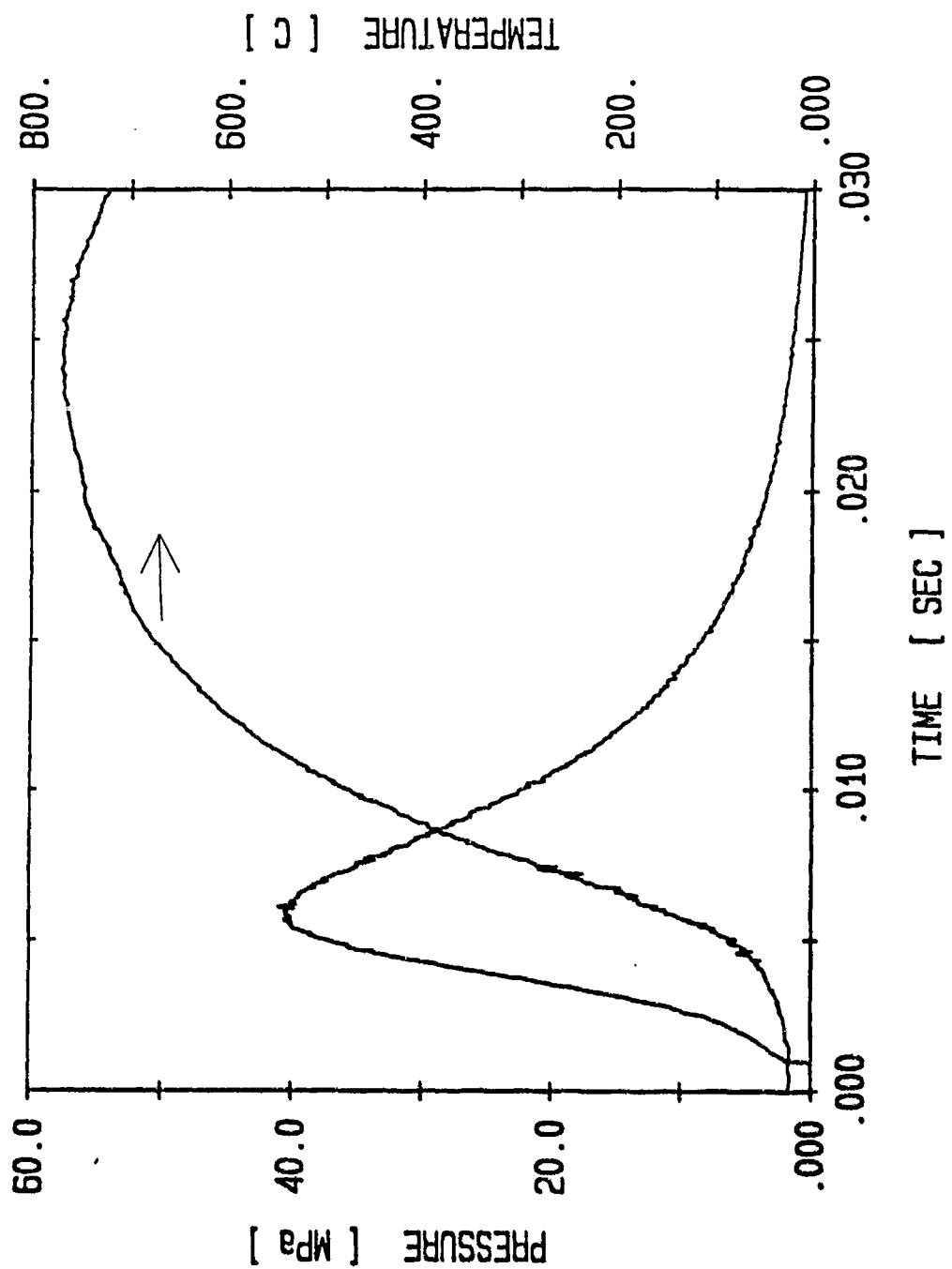


Figure A-6. Igniting-Charge Test Fixture Data for Goex Black Powder [Run 53]

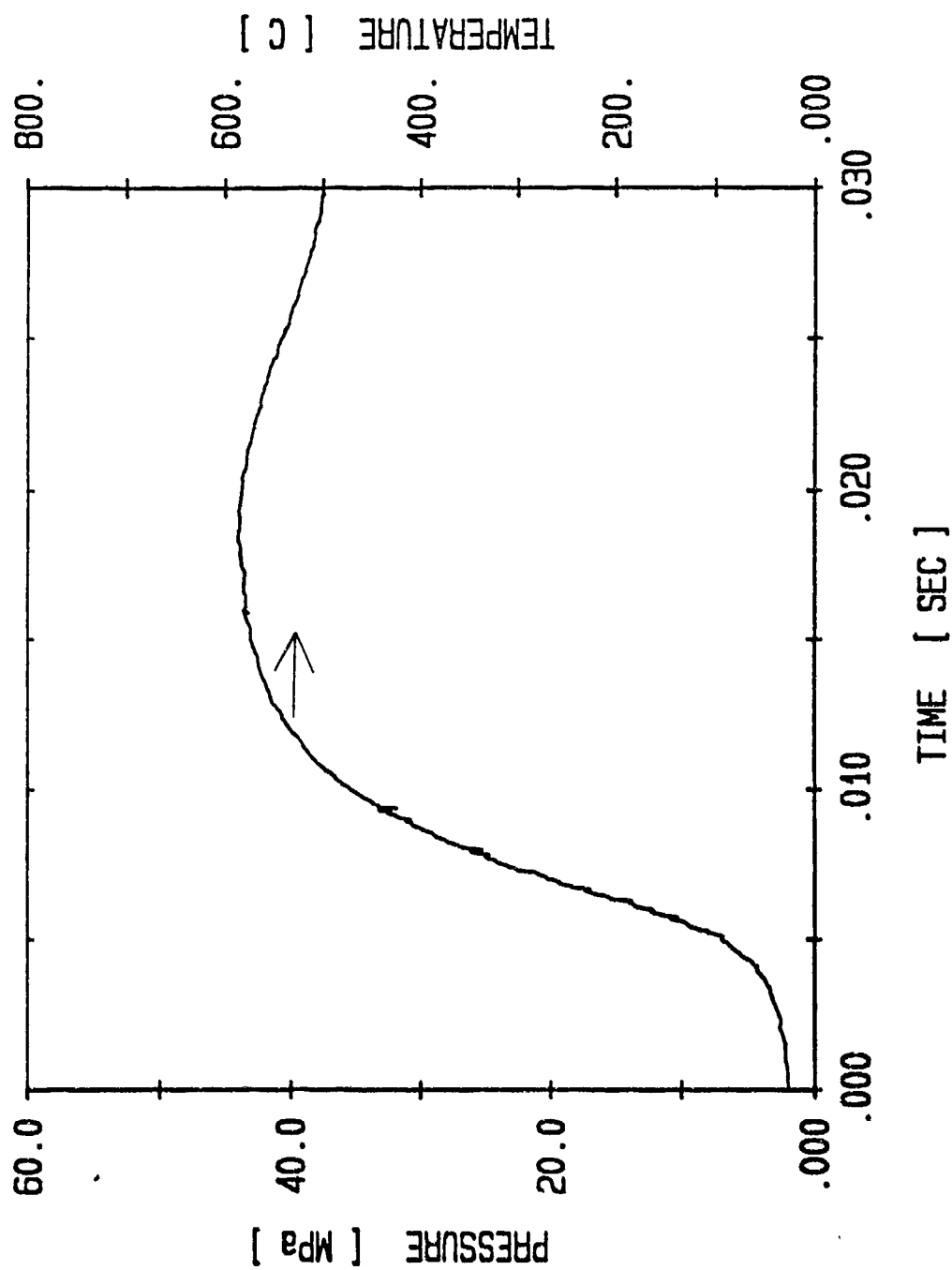


Figure A-7. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 59]

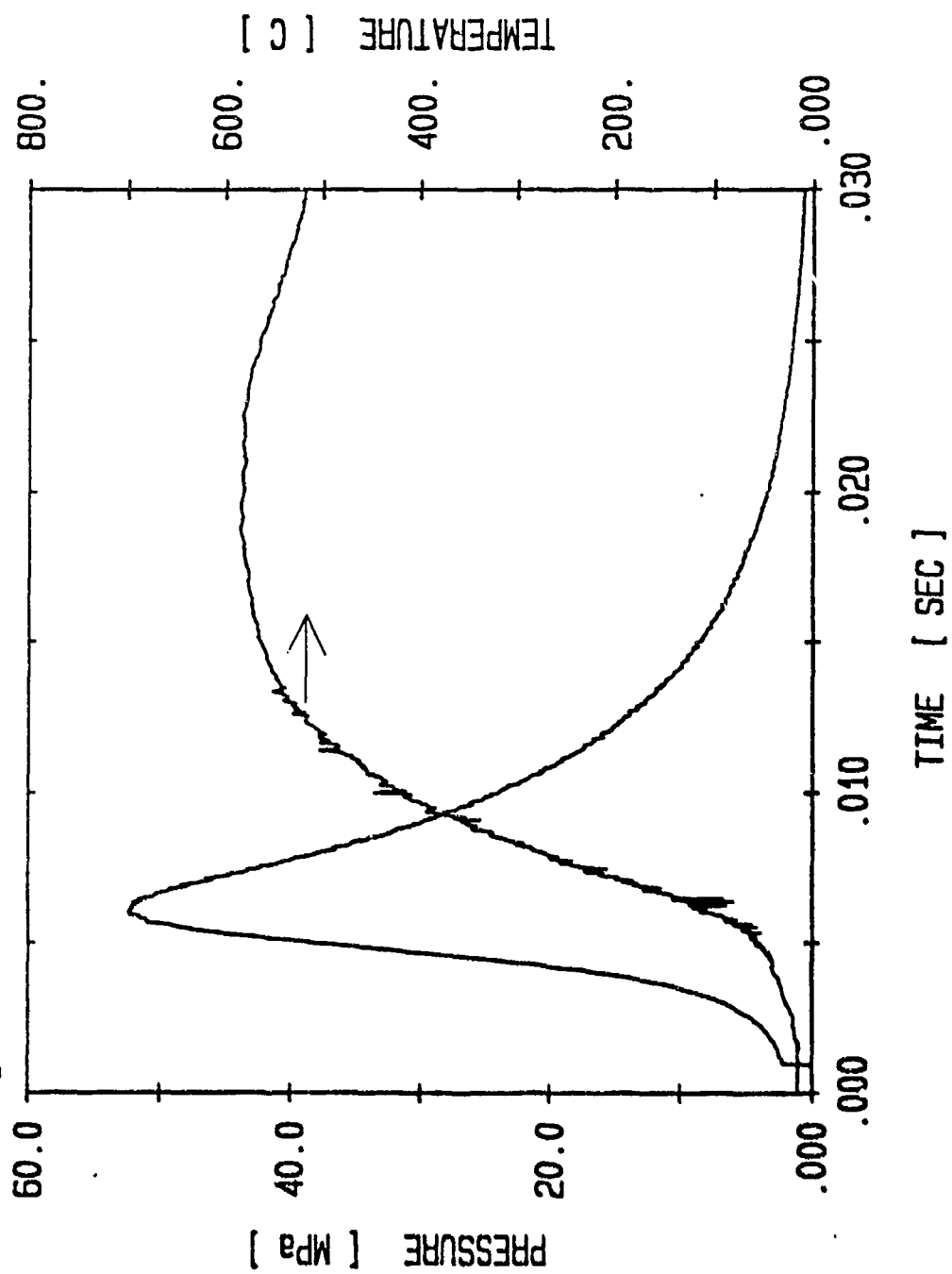


Figure A-8. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 60]

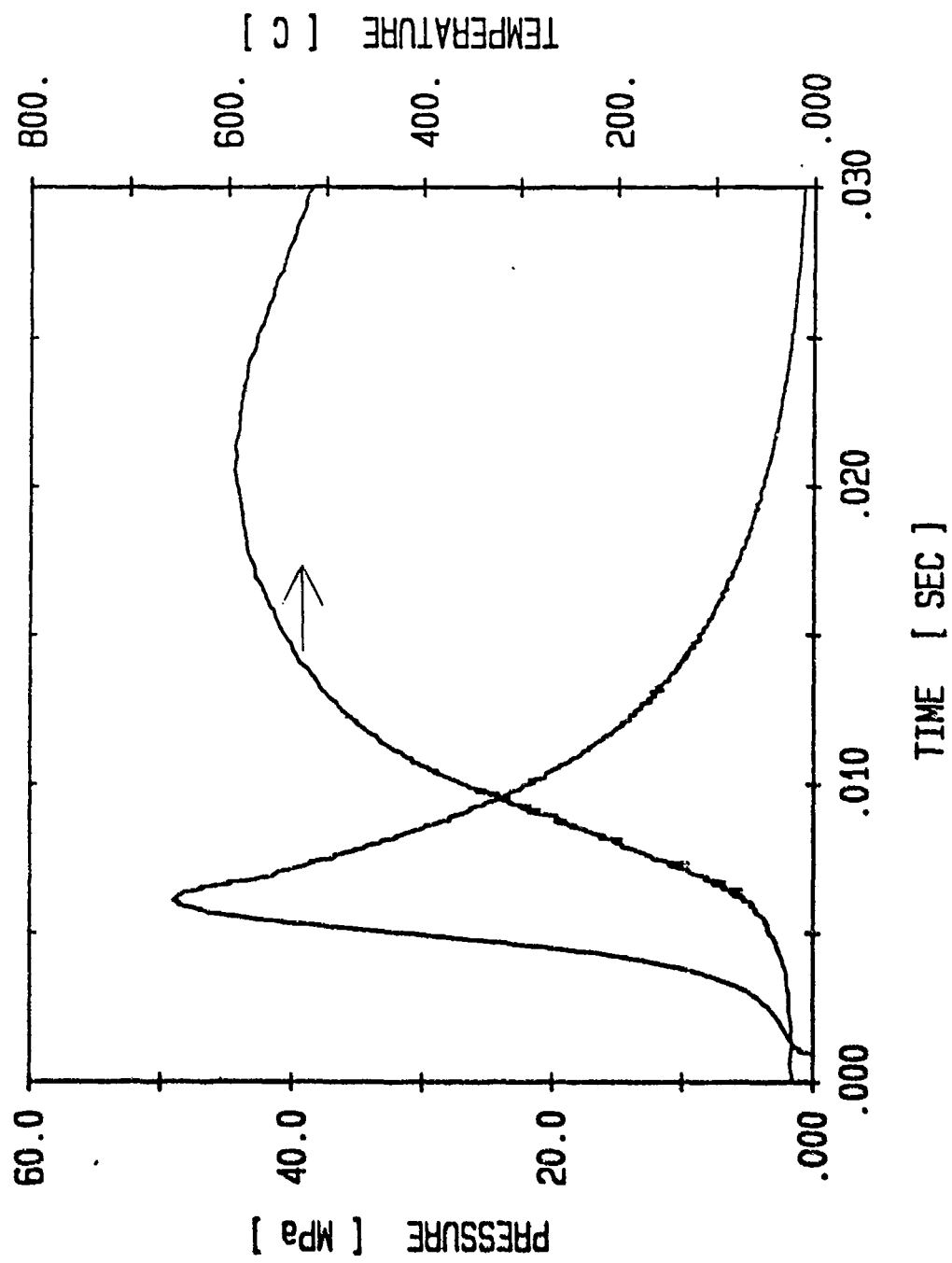


Figure A-9. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 61]

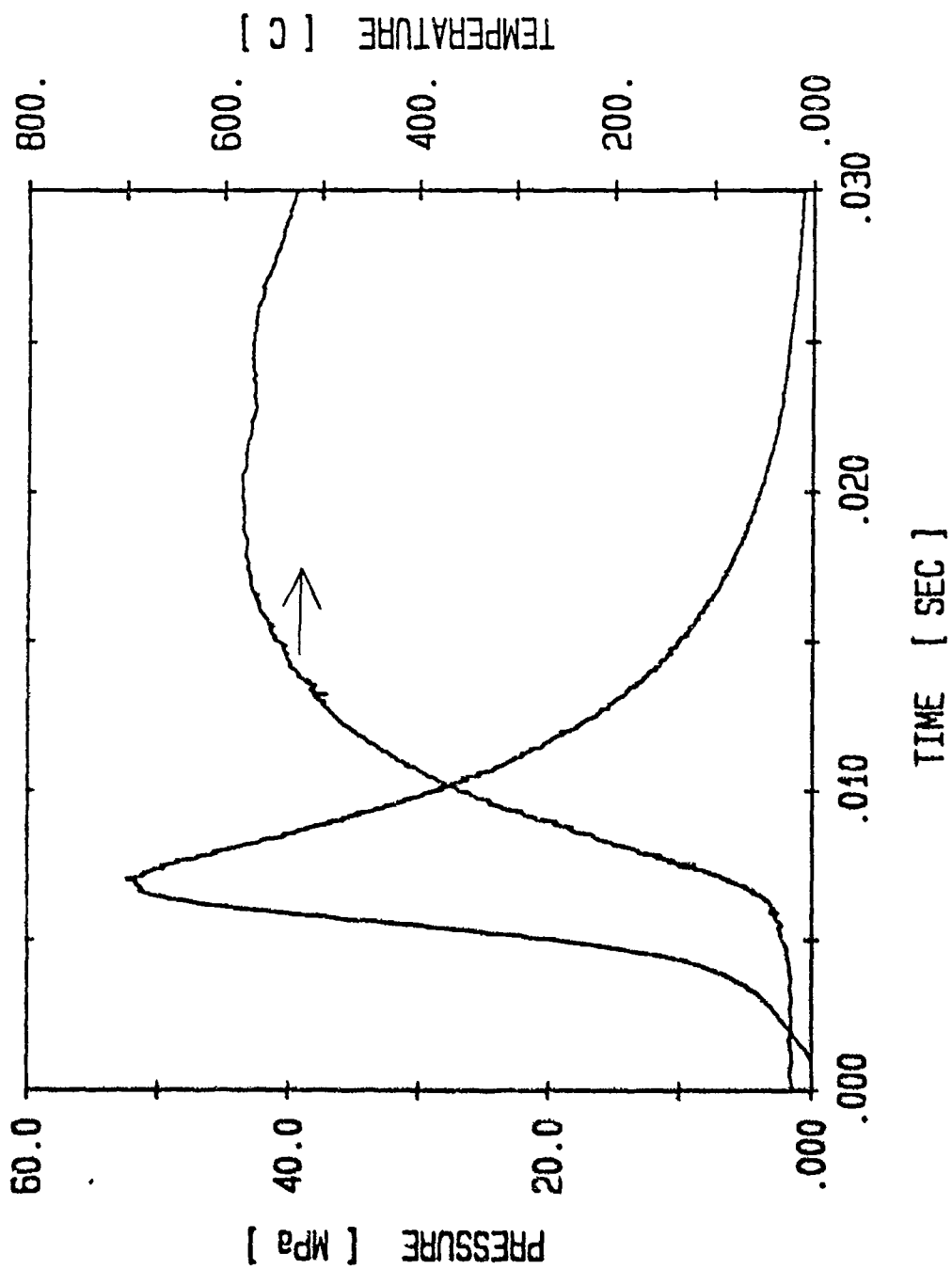


Figure A-10. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 62]

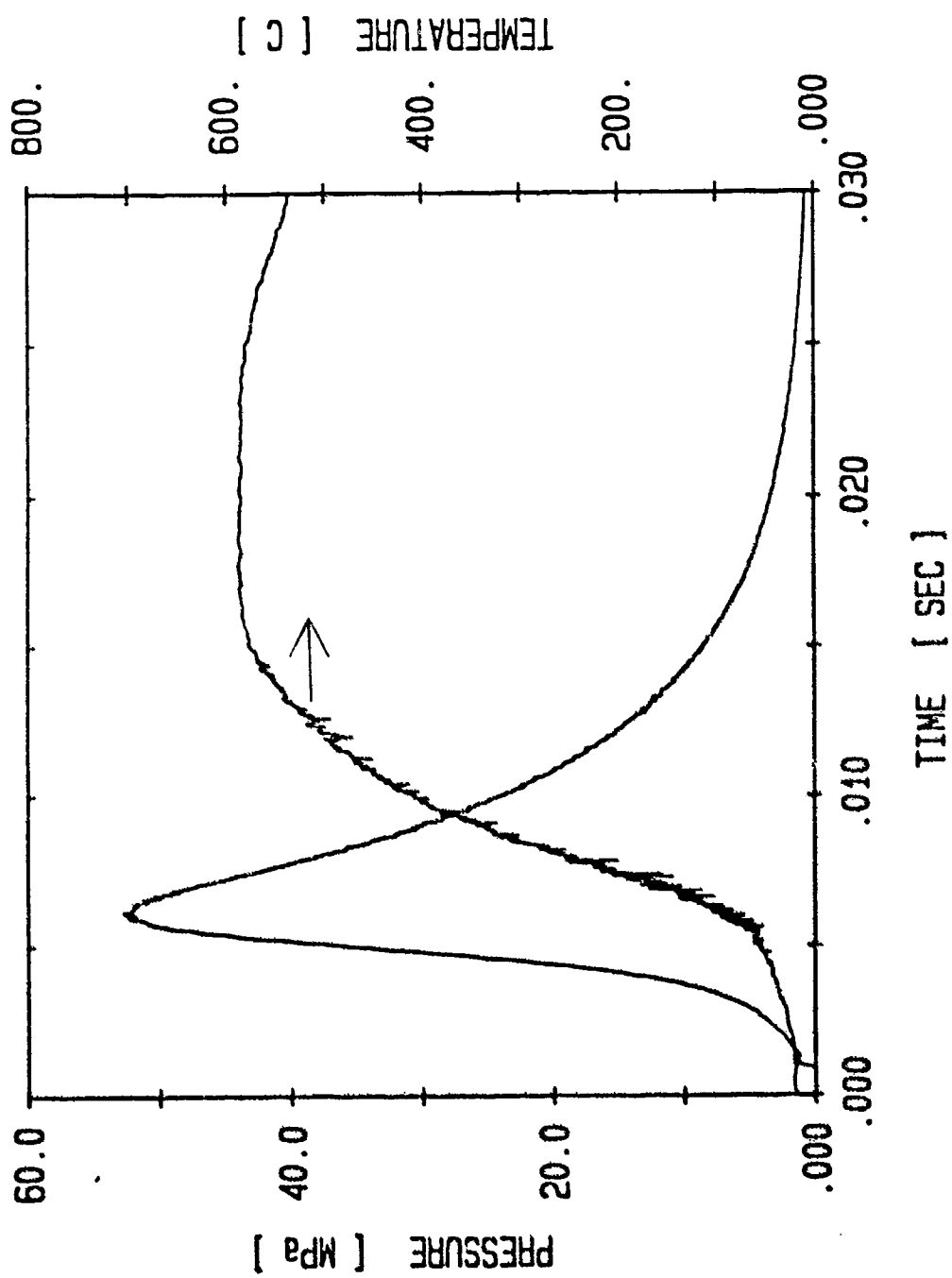


Figure A-11. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 63]

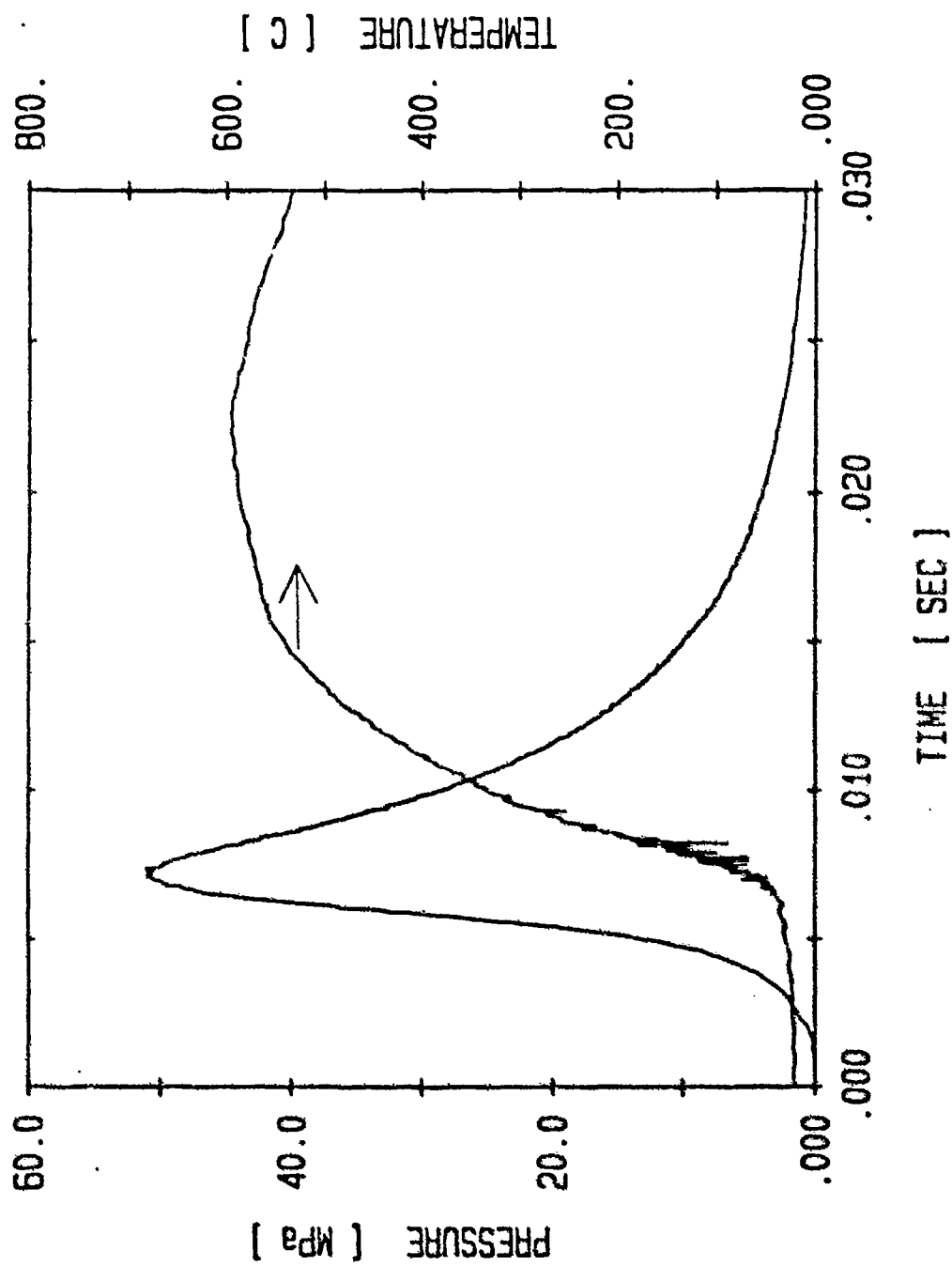


Figure A-12. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 64]

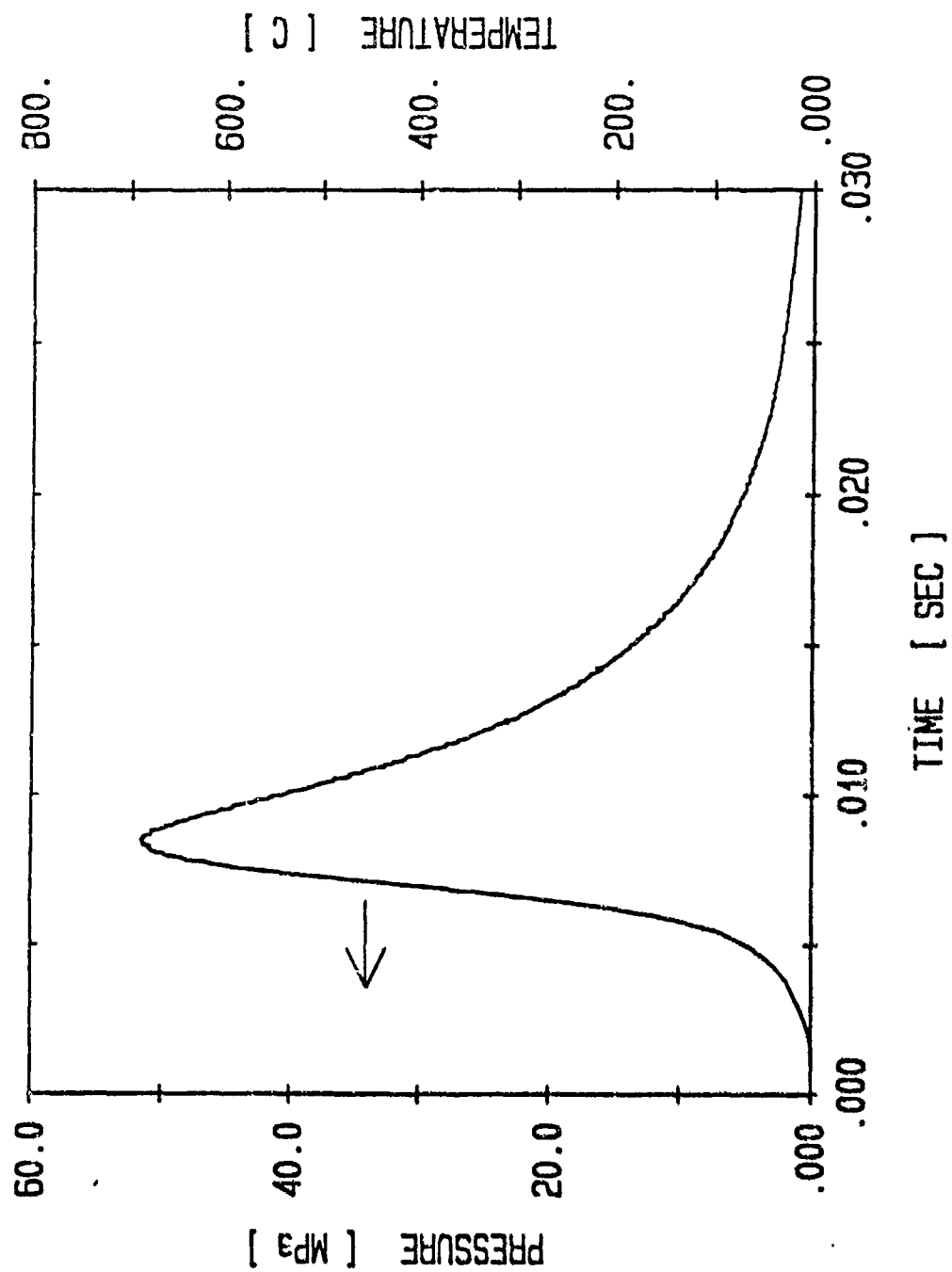


Figure A-13. Igniting-Charge Test Fixture Data for Pyrodex RS [Run 65]

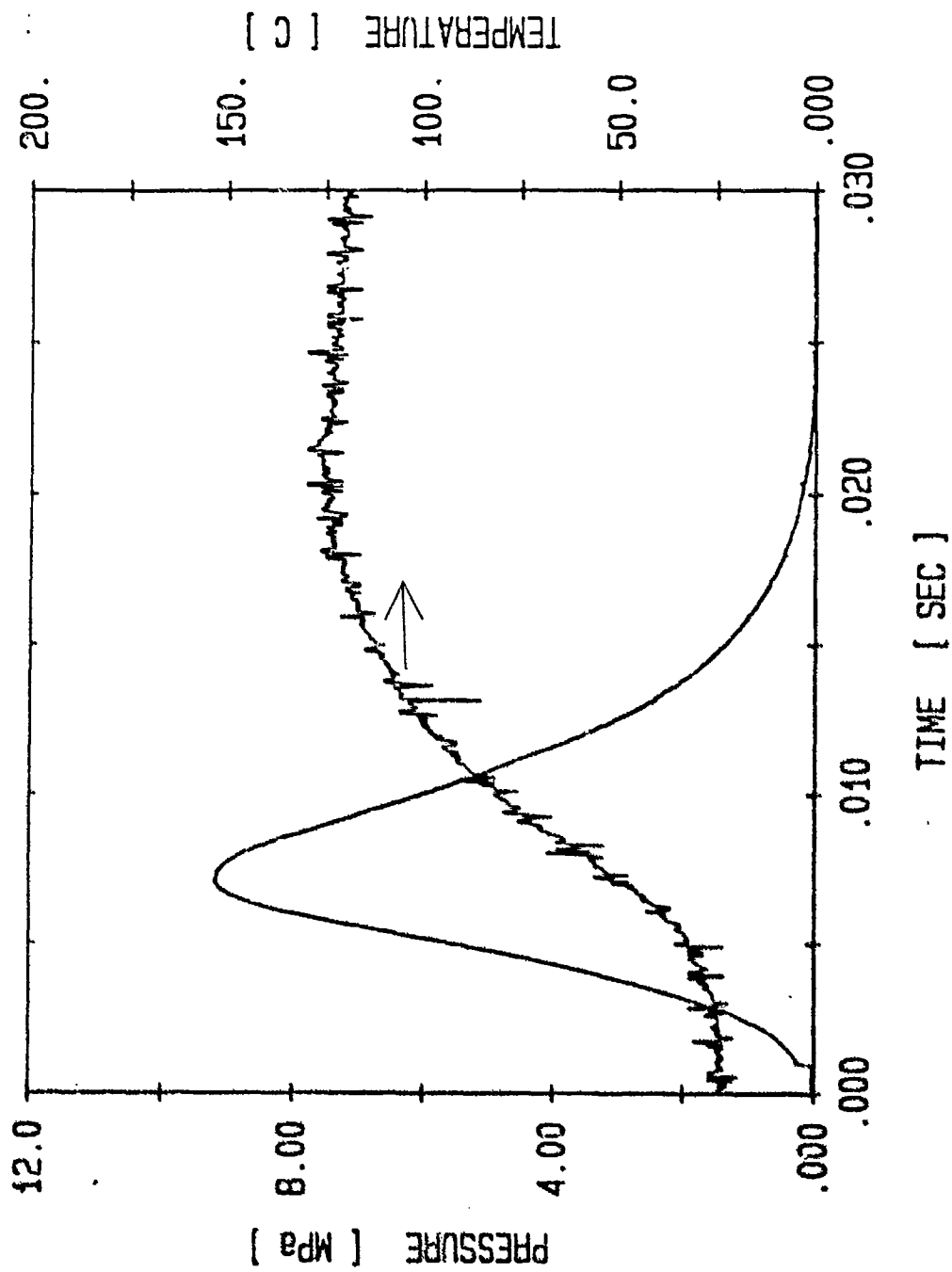


Figure A-14. Expelling-Charge Test Fixture Data for Goex Black Powder [Run 34]

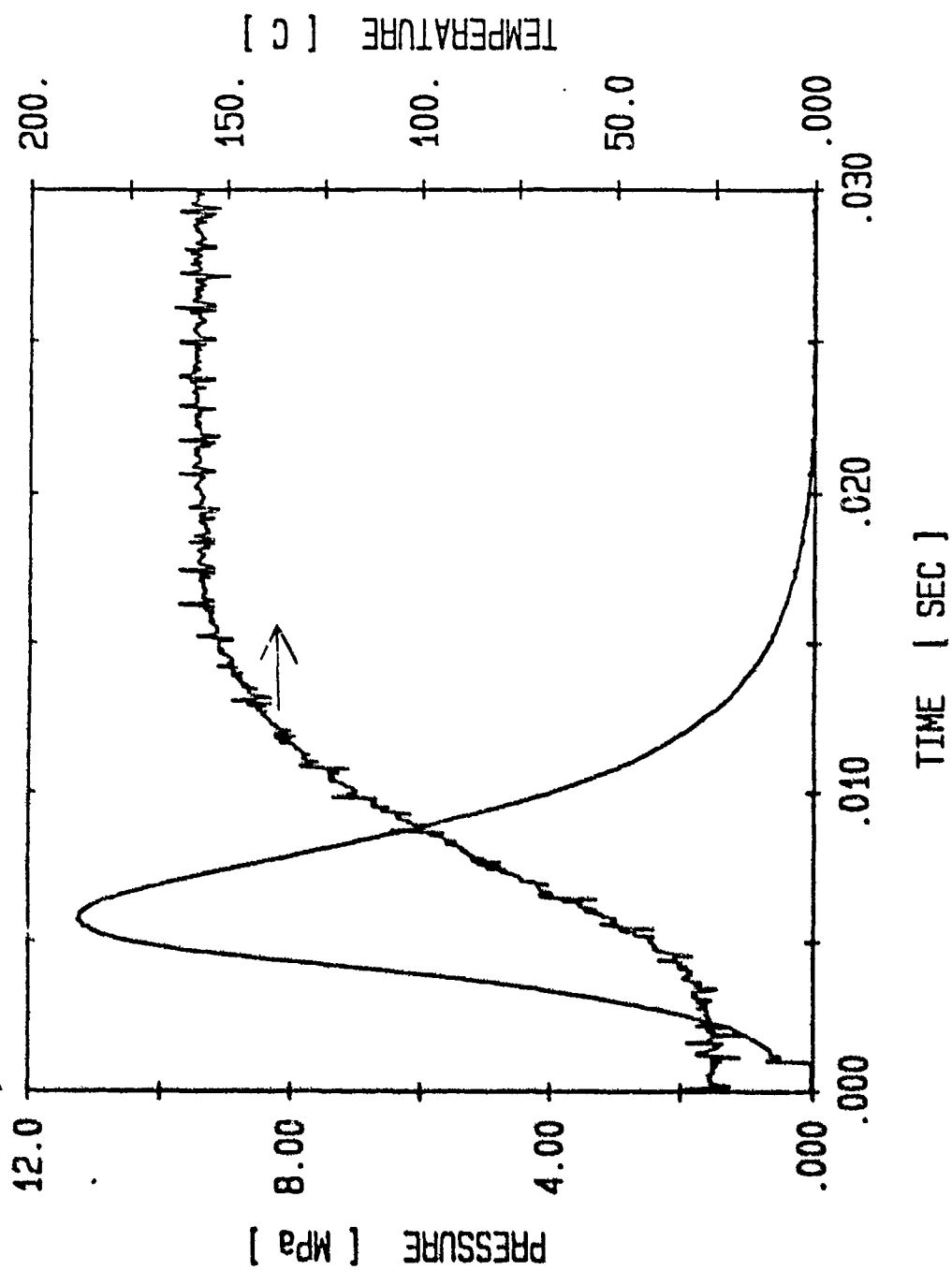


Figure A-15. Expelling-Charge Test Fixture Data for Goex Black Powder [Run 35]

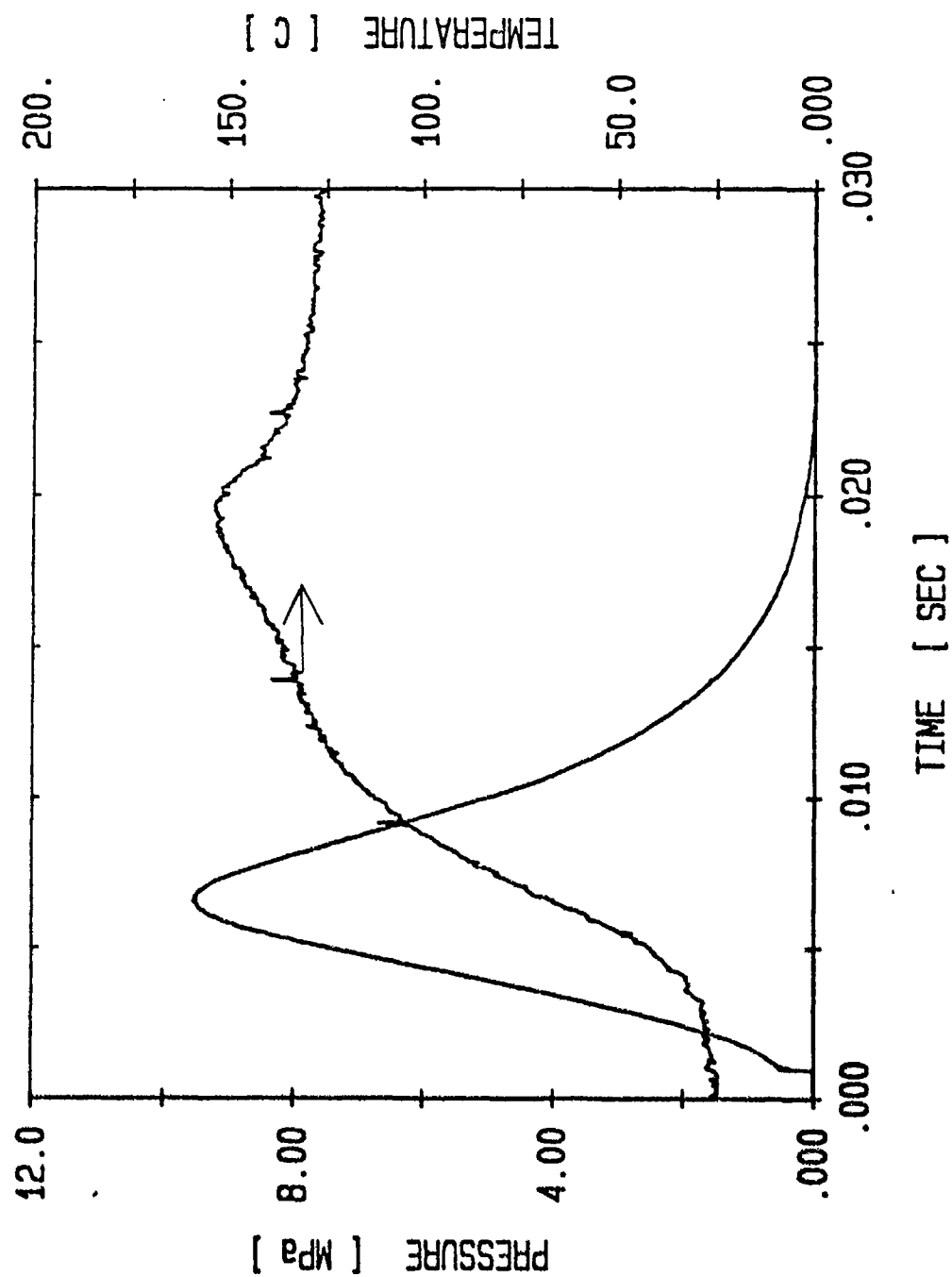


Figure A-16. Expelling-Charge Test Fixture Data for Goex Black Powder [Run 36]

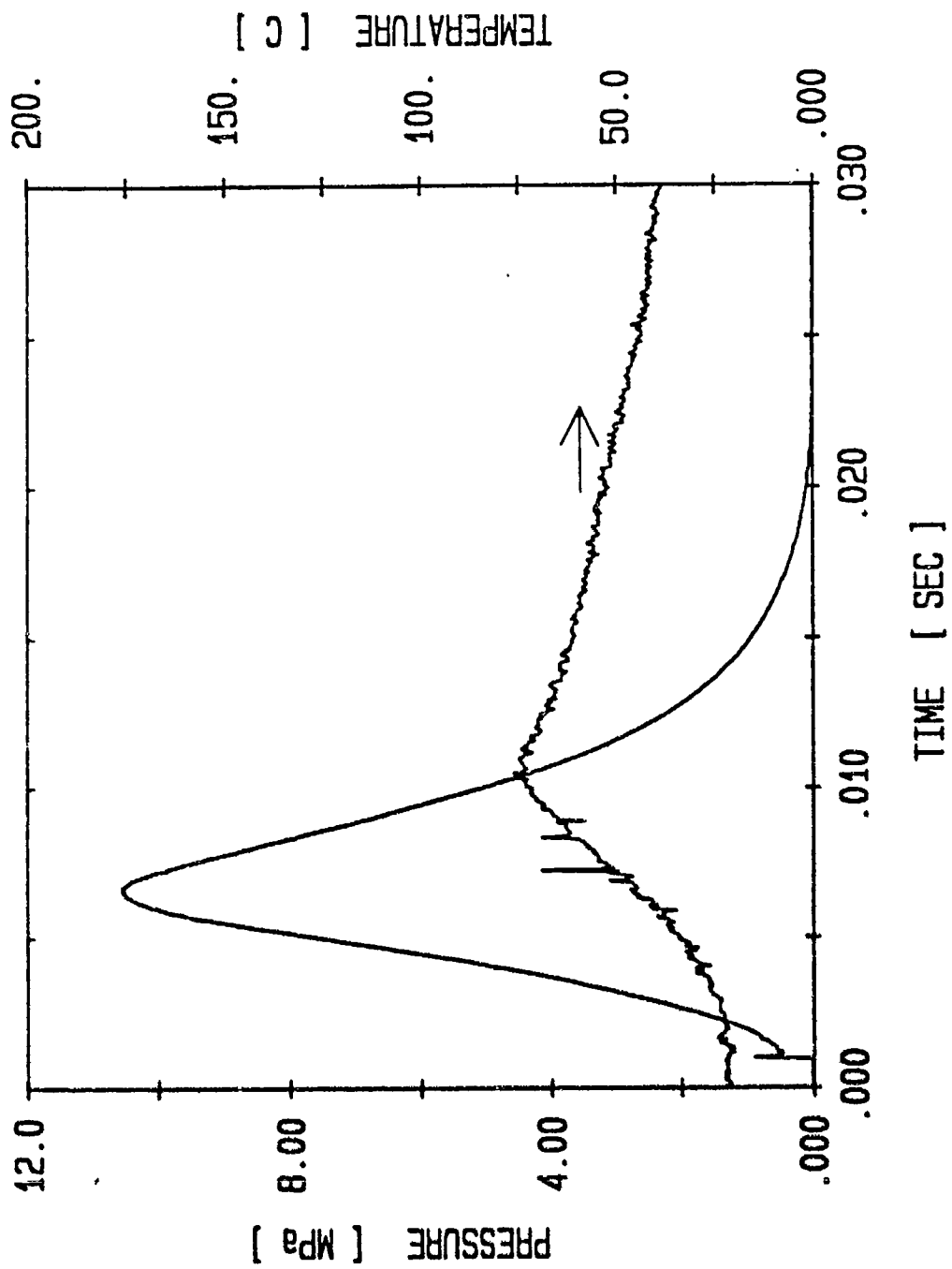


Figure A-17. Expelling-Charge Test Fixture Data for Goex Black Powder [Run 37]

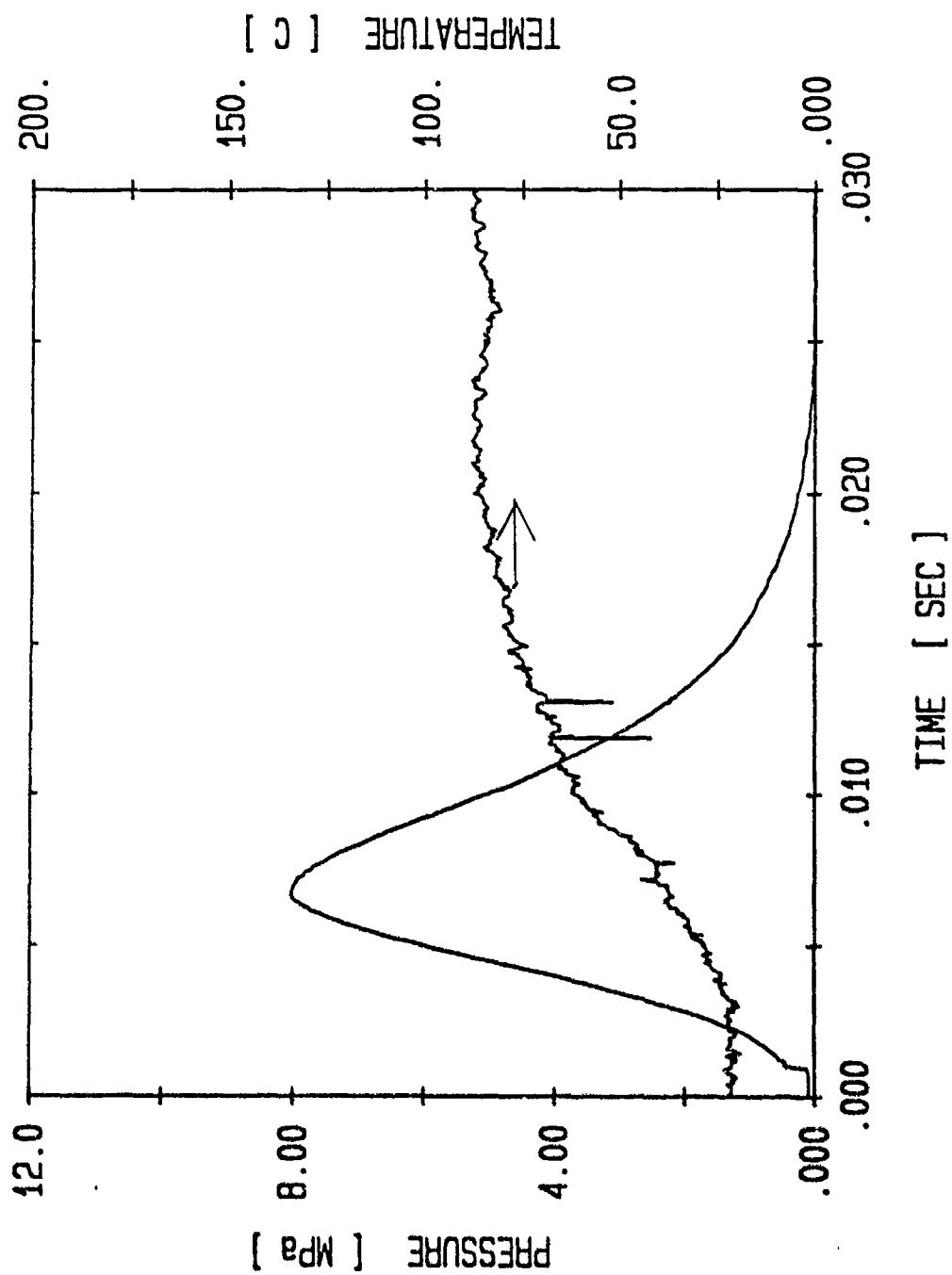


Figure A-18. Expelling-Charge Test Fixture Data for Goex Black Powder [Run 73]

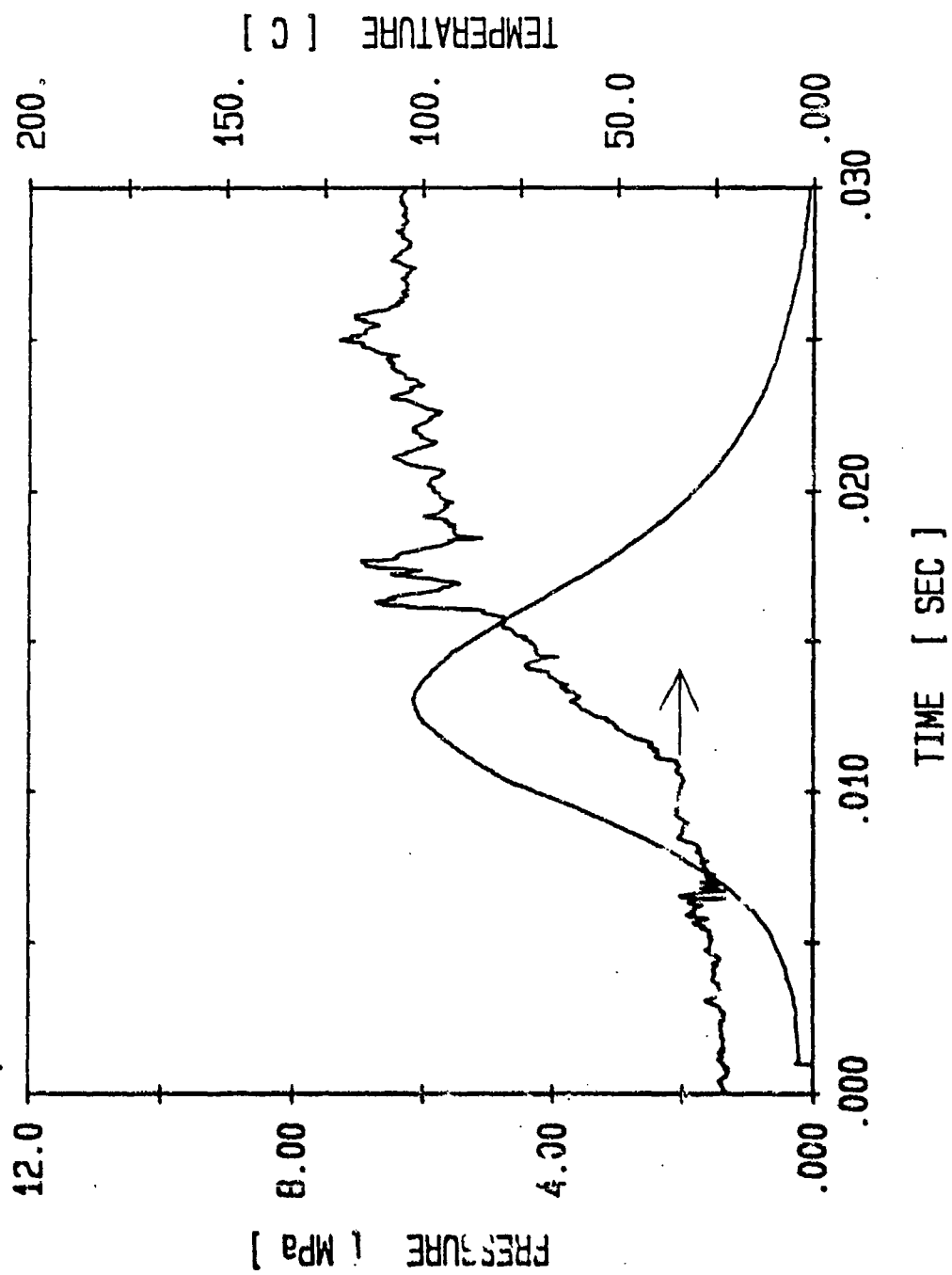


Figure A-19. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 66]

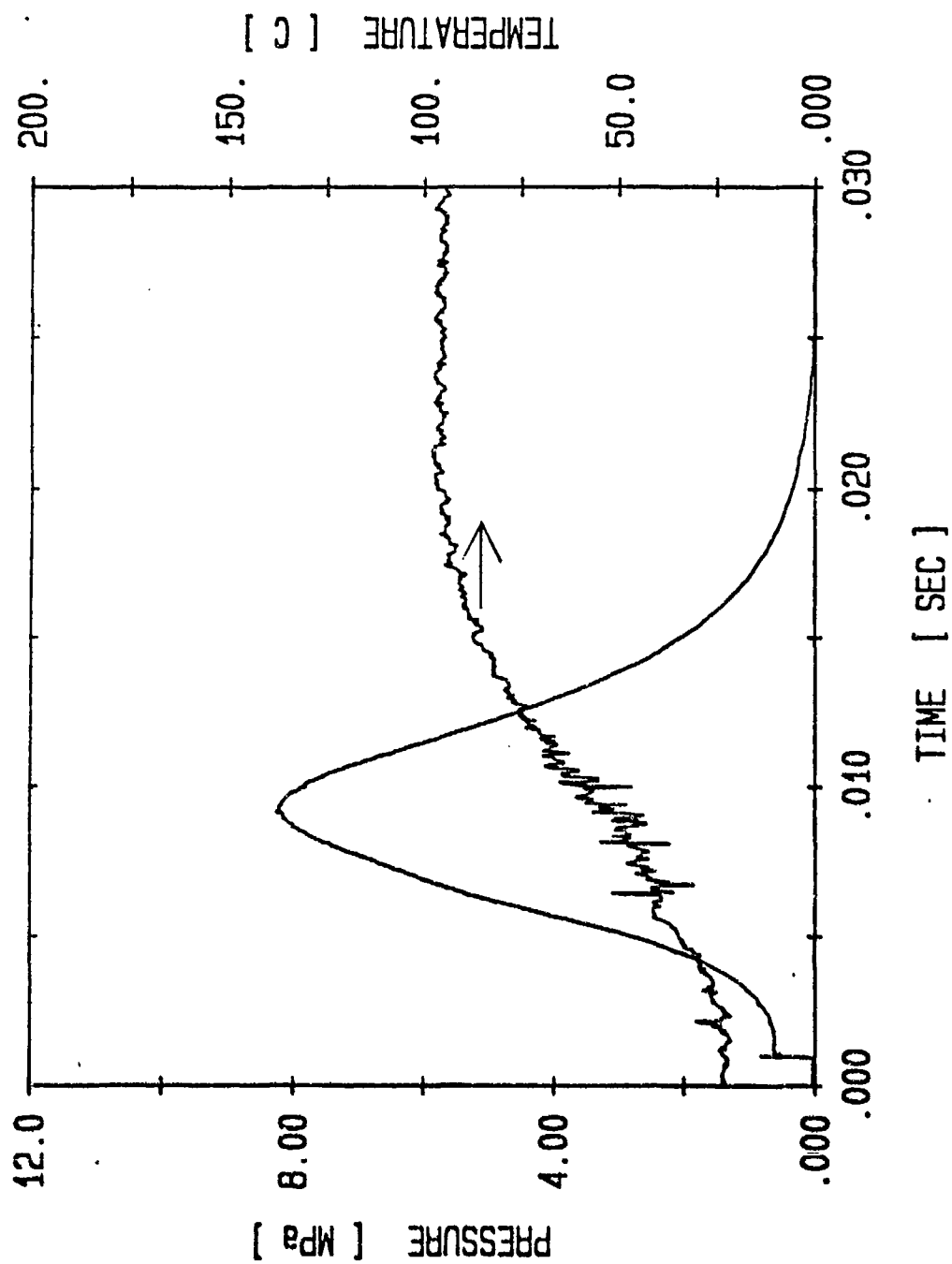


Figure A-20. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 67]

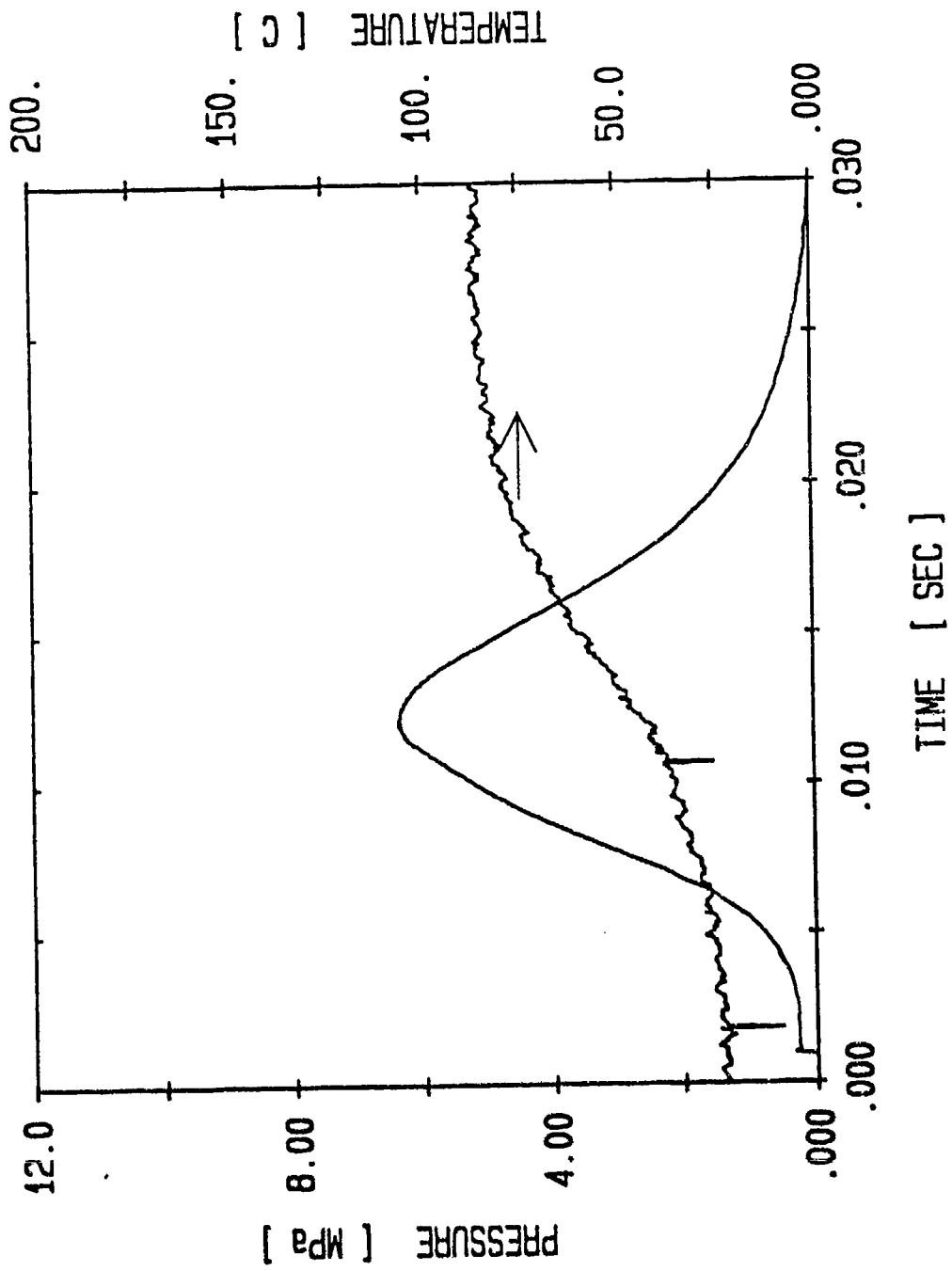


Figure A-21. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 68]

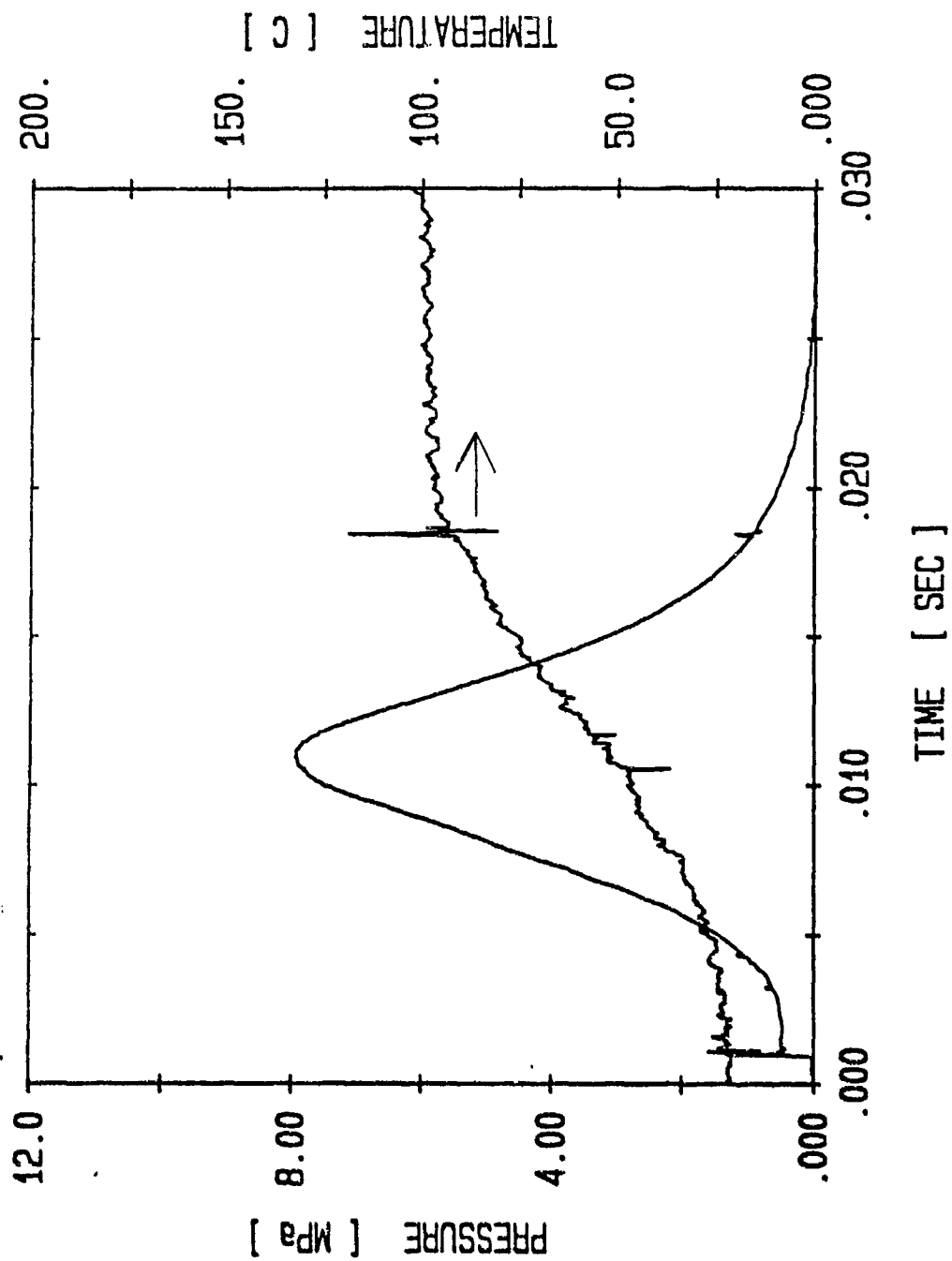


Figure A-22. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 69]

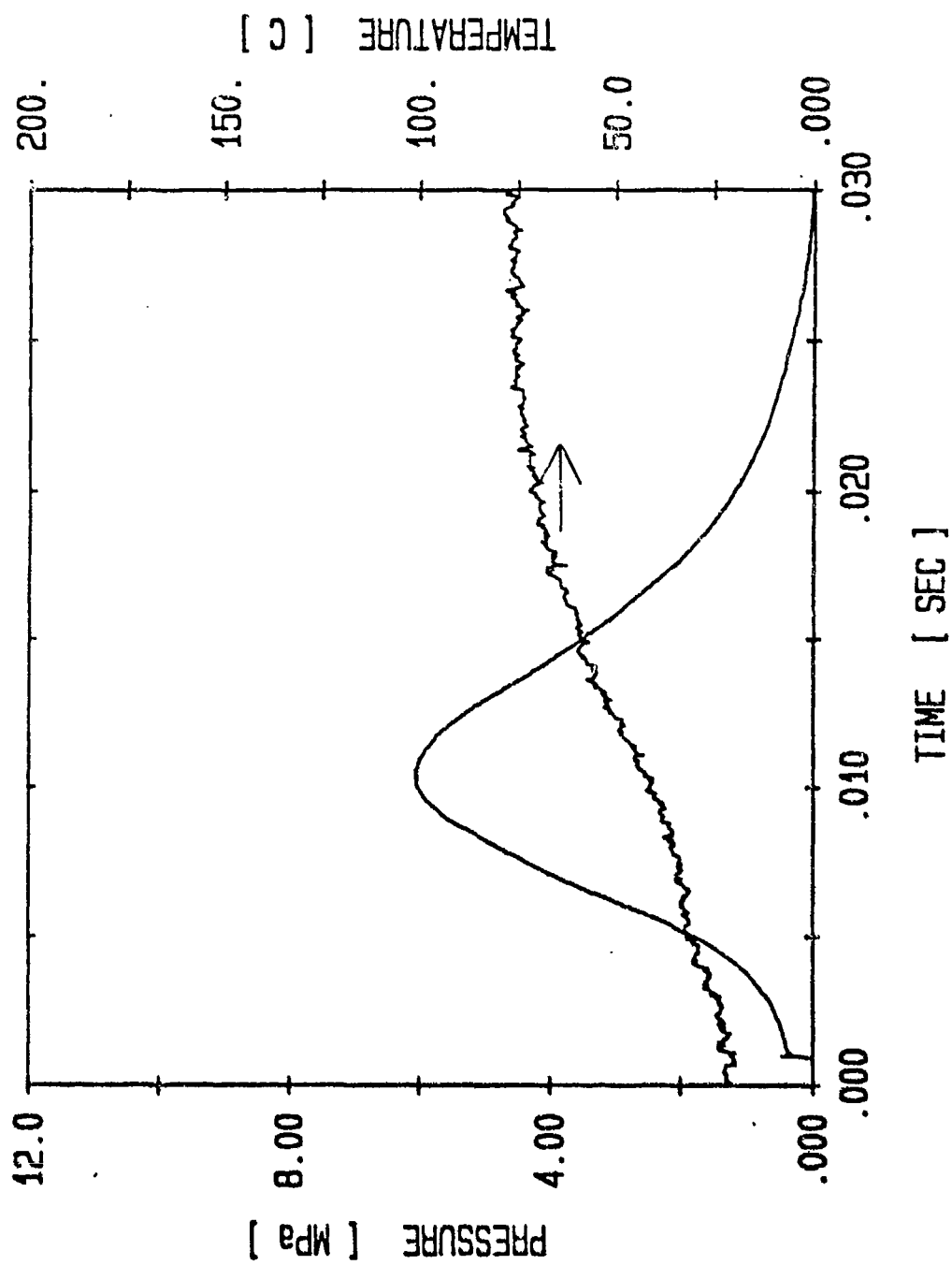


Figure A-23. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 70]

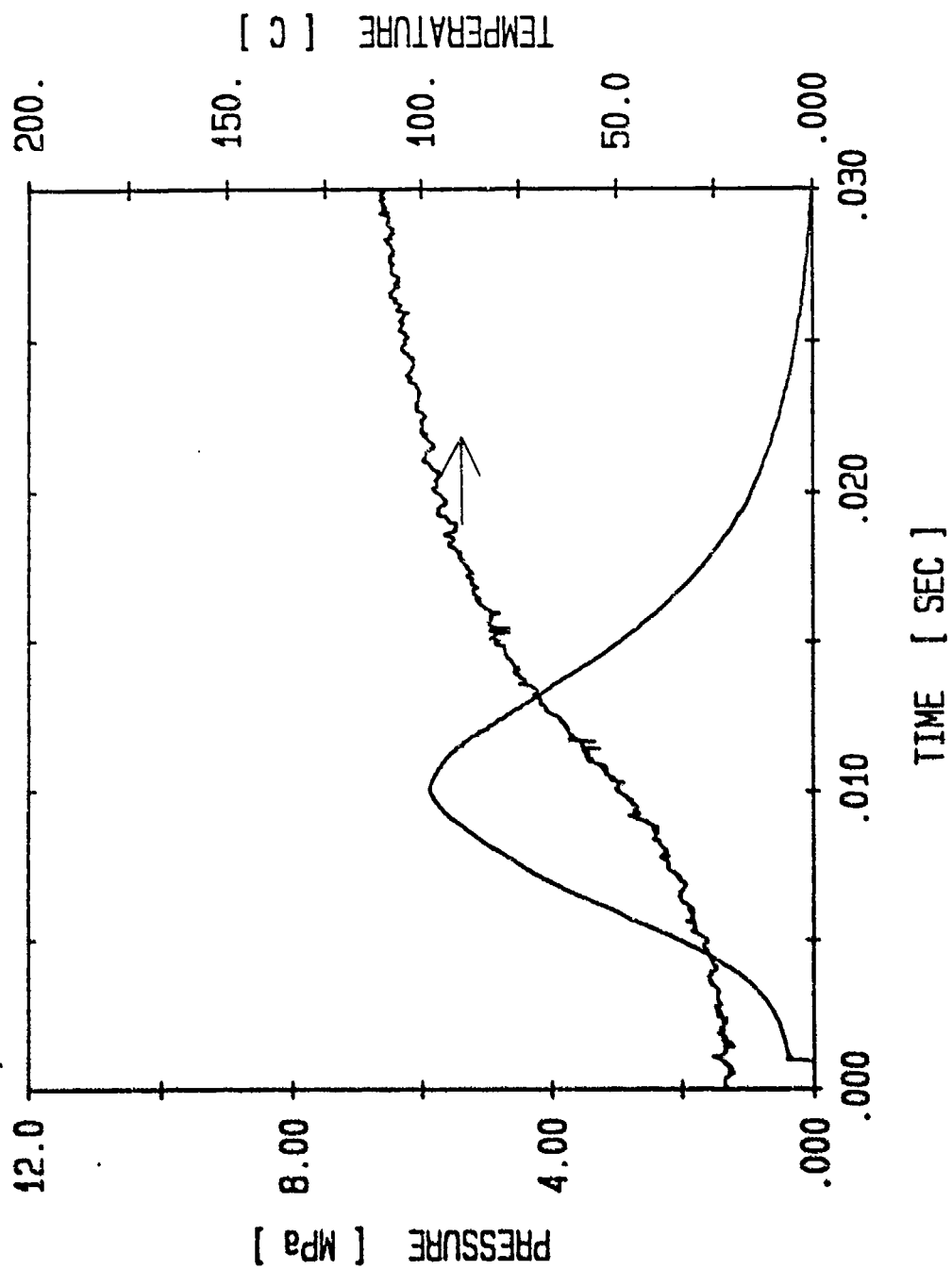


Figure A-24. Expelling-Charge Test Fixture Data for Pyrodex RS [Run 71]

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